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Method 25: Determination of Total Gaseous Non-Methane Organic Emissions as Carbon from Stationary Sources

Deeco, Inc., Cary, NC

Prepared for:

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16. ABSTRACT

Section 3.17 describes the procedures and specifications for determining volatile organic compounds as total gaseous non-methane organics from stationary sources. An emission sample is withdrawn from the stack at a constant rate through a heated filter and a chilled condensate trap by means of an evacuated sample tank. After sampling is completed, the TGNMO are determined by independently analyzing the condensate trap and sample tank fractions and combining the analytical results. The organic content of the condensate trap fraction is determined by oxidizing the NMO to CO_2 and quantitatively collecting the effluent in an evacuated vessel; then a portion of the CO_2 is reduced to CH_4 and measured by an FID. The organic content of the sample tank fraction is measured by injecting a portion of the sample into a gas chromatographic column to separate the NMO from carbon monoxide (CO), CO_2, and CH_4; the NMO are oxidized to CO_2, reduced to CH_4, and measured by an FID. Quality assurance guidance is provided in Section 3.17.

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Section 3.17

METHOD 25 - DETERMINATION OF TOTAL GASEOUS NONMETHANE ORGANIC EMISSIONS AS CARBON FROM STATIONARY SOURCES

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SUMMARY

Method 25 applies to the measurement of volatile organic compounds (VOC) as total gaseous nonmethane organics (TGNMO) as carbon in source emissions. Organic particulate matter will interfere with the analysis and, therefore, a particulate filter is required. The minimum detectable concentration for the method is 50 ppm as carbon.

When carbon dioxide (CO_2) and water vapor are present together in the stack, they can produce a positive bias in the sample. The magnitude of the bias depends on the concentrations of CO_2 and water vapor. As a guideline, multiply the CO_2 concentration times the water vapor concentration, both expressed as volume percent. If this product does not exceed 100, the bias can be considered insignificant. For example, the bias is not significant for source emissions containing 10 percent CO_2 and 10 percent water vapor, but it would be significant for a source with VOC emissions near the detection limit and with 10 percent CO_2 and 20 percent water vapor.

An emission sample is withdrawn from the stack at a constant rate through a heated filter and a chilled condensate trap by means of an evacuated sample tank. After sampling is completed, the TGNMO are determined by independently analyzing the condensate trap and sample tank fractions and combining the analytical results. The organic content of the condensate trap fraction is determined by oxidizing the NMO to CO_2 and quantitatively collecting the effluent in an evacuated vessel; then a portion of the CO_2 is reduced to CH_4 and measured by a flame ionization detector (FID). The organic content of the sample tank fraction is measured by injecting a portion of the sample into a gas chromatographic column to separate the NMO from carbon monoxide (CO), CO_2, and CH_4; the NMO are oxidized to CO_2, reduced to CH_4, and measured by an FID. In this manner, the variable response of the FID associated with different types of organics is eliminated.

This method is not the only method that applies to the measurement of TGNMO. Costs, logistics, and other practicalities of source testing may make other test methods more desirable for measuring VOC contents of certain effluent streams. Proper judgment is required in determining the most applicable VOC test method. For example, depending upon the molecular weight of the organics in the effluent stream, a totally automated semicontinuous nonmethane organics (NMO) analyzer interfaced directly to the source may yield accurate results. This approach has the advantage of providing emission data semicontinuously over an extended time period.

Direct measurement of an effluent with an FID analyzer may be appropriate with prior characterization of the gas stream and knowledge that the detector responds predictably to the organic compounds in the stream. If present, methane (CH_4) will, of course, also be measured. The FID can be applied to the determination of the mass concentration of the total molecular structure of the organic emissions under any of the following limited conditions: (1) where only one compound is known to exist; (2) when the organic compounds consist of only hydrogen and carbon; (3) where the relative percentages of the compounds are known or can be determined, and the FID responses to the compounds are known; (4) where a consistent mixture of the compounds exists before and after emission control and only the relative concentrations are to be assessed; or (5) where the FID can be calibrated against mass standards of the compounds emitted (solvent emissions, for example).

Another example of direct use of an FID is as a screening method. If there is enough information available to provide a rough estimate of its accuracy, the FID analyzer can be used to determine the VOC content of an uncharacterized gas stream.

With a sufficient buffer to account for possible inaccuracies, direct use of an FID can be a useful tool to obtain the desired measurements without costly exact determination. In situations where qualitative/quantitative analysis of an effluent stream is desired or required, a gas chromatographic FID system may apply. However, for sources emitting numerous organics, the time and expense of this approach will be formidable.

METHOD HIGHLIGHTS

Section 3.17 describes the procedures and specifications for determining volatile organic compounds as total gaseous nonmethane organics from stationary sources. An emission sample is withdrawn from the stack at a constant rate through a heated filter and a chilled condensate trap by means of an evacuated sample tank. After sampling is completed, the TGNMO are determined by independently analyzing the condensate trap and sample tank fractions and combining the analytical results. The organic content of the condensate trap fraction is determined by oxidizing the NMO to CO_2 and quantitatively collecting the effluent in an evacuated vessel; then a portion of the CO_2 is reduced to CH_4 and measured by an FID. The organic content of the sample tank fraction is measured by injecting a portion of the sample into a gas chromatographic column to separate the NMO from carbon monoxide (CO), CO_2, and CH_4; the NMO are oxidized to CO_2, reduced to CH_4, and measured by an FID. In this manner, the variable response of the FID associated with different types of organics is eliminated.

On October 3, 1980 (45 FR 65956), EPA published Method 25, "Determination of Total Gaseous Nonmethane Organic Emissions as Carbon". Shortly after publication, testers began to report erratic results with the method and suggested a number of different causes for the imprecision. As a result, EPA began a program to review the test method in March 1982. The EPA completed the review and proposed revisions to Method 25, designed to make the method simpler, more reliable, and more precise. The results of the various studies on Method 25 are presented in the documents listed in Reference Subsection 3.17.11 (References 1 through 8).

On February 12, 1988, several changes were made to Method 25. The studies had shown that the basic operating principle of Method 25 was sound, but some changes in equipment design and operating practices would improve the reliability of the method. These changes can be discussed by dividing the method into three parts: Sampling, sample recovery, and analysis.

The major changes in the sampling equipment are the addition of a heated filter, a redesigned condensate trap, and a different packing material for the condensate trap. The purpose of the heated filter is to remove organic particulate matter from the sample and, thus, eliminate a potential source of imprecision. It is heated to a temperature of 120 °C (248 °F). The new trap design is a simple U-tube which may be more easily and cheaply produced than the previous design. It also provides a faster and more complete sample recovery than the existing trap while showing equal collection efficiency. The new packing material is quartz wool, which, compared to the previously specified stainless steel packing, is more durable and has improved collection efficiency.

The major changes in the sample recovery are a new oxidation catalyst, a simplified recovery system, and lower operating temperatures. The new oxidation catalyst has proven to be very durable and to provide 100 percent oxidation efficiency for a wide variety of organic compounds at much lower operating temperatures than the old catalyst. The redesigned recovery system has eliminated some of the tubing and valving and, thus, reduced the potential for sample loss during recovery and decreased the recovery time. The lower temperatures for sample recovery will increase the life expectancy of the recovery system materials and simplify the operation of the system.

The major change in the sample analysis system is a new separation column for the nonmethane organics analyzer. This new column provides separation of CO, CO_2, and CH_4 from a wider range of organic compounds than the previously specified column.

In addition to these major changes, there are a number of minor changes, particularly in the areas of quality assurance (QA) and calibration.

Collaborative testing of Method 25 has not been performed. However, results for analysis of performance audit samples have shown that the revised method can meet the required relative error of ± 20 percent of the actual concentration of the audit gas.

The blank data forms may be removed from the Handbook and used in the pretest, on-site, and posttest operations. The items/parameters that can cause the most significant errors are designated with an asterisk.

1. Procurement of Apparatus and Supplies

Section 3.17.1 (Procurement of Apparatus and Supplies) gives specifications, criteria, and design features for the required equipment and materials. This section can be used as a guide for procurement and initial checks of equipment and supplies. The activity matrix (Table 1.1) at the end of the section is a summary of the details given in the text and can be used as a quick reference.

2. Pretest Preparations

Section 3.17.2 (Calibration of Apparatus) describes the required calibration procedures and considerations for the Method 25 sampling equipment. Required accuracies for each component are also included. A pretest checklist (Figure 3.1 in Subsection 3.17.3) or a similar form should be used to summarize the calibration and other pertinent pretest data. The calibration section may be removed along with the corresponding sections for the other methods and made into a separate quality assurance reference manual for use by personnel involved in calibration activities.

Section 3.17.3 (Presampling Operations) provides the tester with a guide for equipment and supplies preparation for the field test. A pretest preparation form can be used as an equipment checkout and packing list. Because of the potential for high blank levels, special attention must be paid in the preparation of the sampling equipment. Also the tester must ensure that the agency obtains the required audit samples for the test.

Activity matrices for the calibration of equipment and the presampling operations (Tables 2.1 and 3.1) summarize the activities.

3. On-Site Measurements

Section 3.17.4 (On-Site Measurements) contains step-by-step procedures for sample collection and sample preparation for transport. The on-site checklist (Figure 4.2, Section 3.17.4) provides the tester with a quick method of checking the on-site requirements. The revised sampling equipment and procedures were designed to help eliminate the contamination of the sample for particulate matter from the source and to provide better collection of condensible organic compounds in the trap and must be closely followed to provide more precise measurements. The audit samples are collected during the field sampling phase. Table 4.1 provides an activity matrix for all on-site activities.

4. Posttest Operations

Section 3.17.5 (Posttest Operations) presents the posttest equipment procedures and a step-by-step analytical procedure for determination of the total nonmethane gaseous organics as carbon. Posttest calibration is not required for any of the sampling equipment. The posttest operations form (Figure 5.1. Section 3.17.5) provides some key parameters to be checked by the tester and laboratory personnel. The step-by-step analytical procedure description can be removed and made into a separate quality assurance analytical reference manual for the laboratory personnel. Initial performance tests of both the condensible organic recovery system and the NMO analyzer must be performed before the systems are first placed into operation, after any shutdown of longer than six months, or after any major modification of the systems. In addition to the initial performance checks, daily performance checks and calibrations must be performed. Analysis of two audit samples is required. Strict adherence to Method 25 analytical procedures must be observed.

Section 3.17.6 (Calculations) provides the tester with the required equations, nomenclature, and significant digits. Because of the complex nature of the method and the large number of checks, an example data reporting format is shown. It is suggested that a calculator or computer be used, if available, to reduce the chances of calculation error.

Section 3.17.7 (Maintenance) provides the tester with a guide for a maintenance program. This program is not required, but should reduce equipment malfunctions. Activity matrices (Tables 5.1, 6.1, and 7.1) summarize all postsampling, calculation, and maintenance activities.

5. Auditing Procedures

Section 3.17.8 (Auditing Procedure) provides a description of necessary activities for conducting performance and system audits. The performance audit of the sampling and analytical phase can be conducted using audit gas cylinders supplied by the Quality Assurance Division, Atmospheric Research and Exposure Assessment Laboratory, U. S. Environmental Protection Agency. The data processing procedures and a checklist for a systems audit are also included in this Section. Table 8.1 is an activity matrix for conducting the performance and system audits.

Section 3.17.9 (Recommended Standards for Establishing Traceability) provides the primary standard to which the analytical data should be traceable.

6. References

Section 3.17.10 contains the promulgated Method 25 and Section 3.17.11 contains the references cited throughout the text.

1.0 PROCUREMENT OF APPARATUS AND SUPPLIES

A schematic of the sampling train used for Method 25 is shown in Figure 1.1 and a schematic of the analytical equipment is shown in Figure 1.2. Specifications, criteria, and/or design features are presented in this section to aid in the selection of equipment. Many of the sampling train components can be manufacturer by the tester or other vendors. Procedures and limits (where applicable) for acceptance checks are also presented. Calibration data generated in the acceptance checks are to be recorded in the calibration log book.

During the procurement of equipment and supplies, it is suggested that a procurement log be used to record the descriptive title of the equipment, identification number (if applicable), and the results of acceptance checks.

The following procedures and descriptions are only provided as guidance to the tester and may not be requirements of the method for the initial ordering and check out of the equipment and supplies. The tester should note that many of these procedures are required at a later step in the sampling and analytical procedures. It is therefore in the best interest of the sampling and analytical firm that these procedures or other similar procedures be instituted as routine practice for checking new equipment and supplies to prevent later problems and/or delays in test programs. Table 1.1 at the end of this section contains a summary of quality assurance activities for procurement and acceptance of apparatus and supplies.

1.1 Sampling

The sampling system consists of a heated probe, heated filter, condensate trap, flow control system, and sample tank (Figure 1.1). The TGNMO sampling equipment can be constructed from commercially available components and components fabricated in a machine shop. Complete sampling systems are commercially available that have been designed to meet all EPA equipment design specifications. The following equipment is required:

1.1.1 *Heated Probe* - 6.4-mm (1/4-in.) outside diameter (OD) stainless steel tubing with a heating system capable of maintaining a gas temperature at the exit end of at least 129°C (265°F). The probe shall be equipped with a thermocouple at the exit end to monitor the gas temperature.

A suitable probe is shown in Figure 1.1. The nozzle is an elbow fitting attached to the front end of the probe while the thermocouple is inserted in the side arm of a tee fitting attached to the rear of the probe. The probe is wrapped with a suitable length of high temperature heating tape, and then covered with two layers of glass cloth insulation and one layer of aluminum foil.

NOTE: If it is not possible to use a heating system for safety reasons, an unheated system with an in-stack filter is a suitable alternative.

Upon receipt or after construction, visually check the probe for problems, and plug in the probe heating system to ensure it will heat. Check the thermocouple at room temperature to ensure it is functional, and check the probe heating system in conjunction with the entire sampling system as described later in Subsection 1.1.10. If desired, it may be checked separately by following the checkout procedures in Subsection 1.1.10 that relate to the sample probe.

1.1.2 *Filter Holder* - 25-mm (15/16-in.) inside diameter (ID) Gelman filter holder or equivalent with stainless steel body and stainless steel support screen with the Viton O-ring replaced by a Teflon O-ring. Upon receipt or after construction,

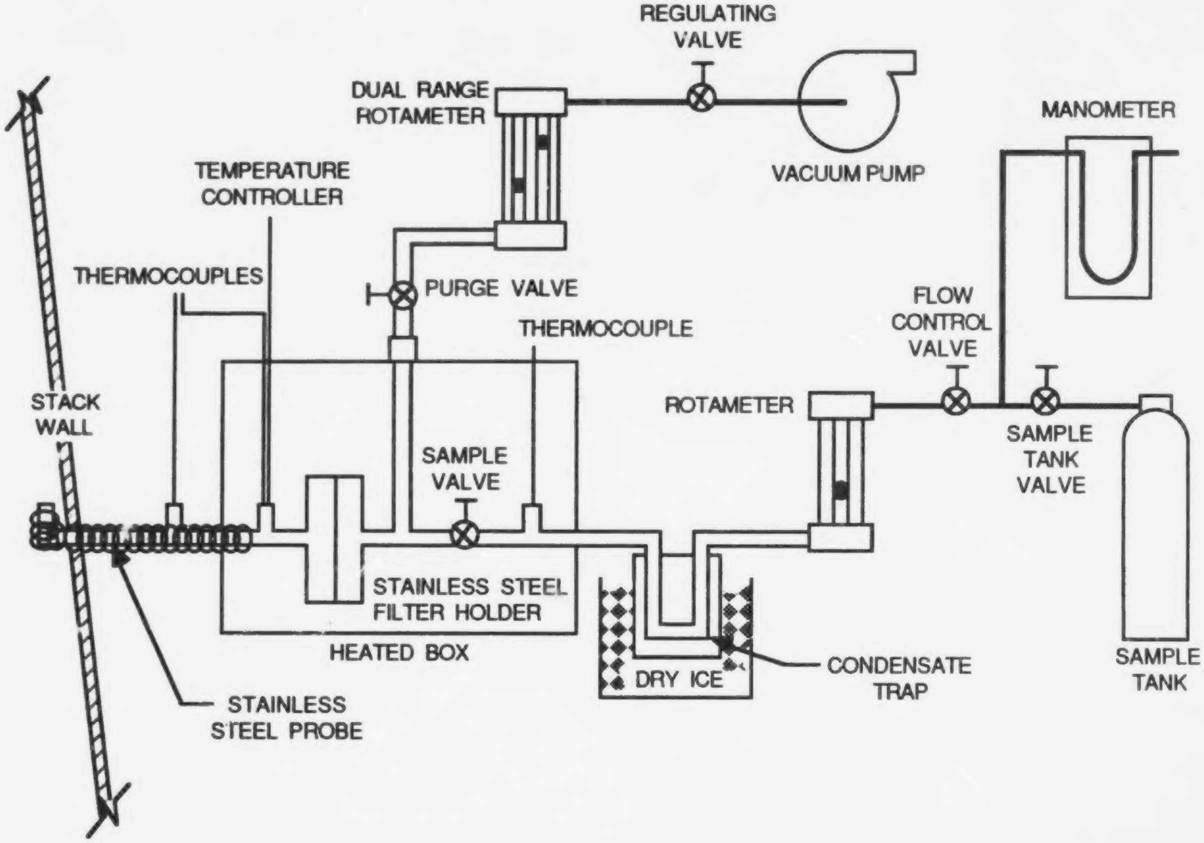
REGULATING VALVE
DUAL RANGE ROTAMETER
VACUUM PUMP
MANOMETER
TEMPERATURE CONTROLLER
THERMOCOUPLES
PURGE VALVE
THERMOCOUPLE
FLOW CONTROL VALVE
STACK WALL
ROTAMETER
SAMPLE VALVE
SAMPLE TANK VALVE
STAINLESS STEEL FILTER HOLDER
HEATED BOX
DRY ICE
CONDENSATE TRAP
SAMPLE TANK
STAINLESS STEEL PROBE

Figure 1.1. Method 25 sampling train.

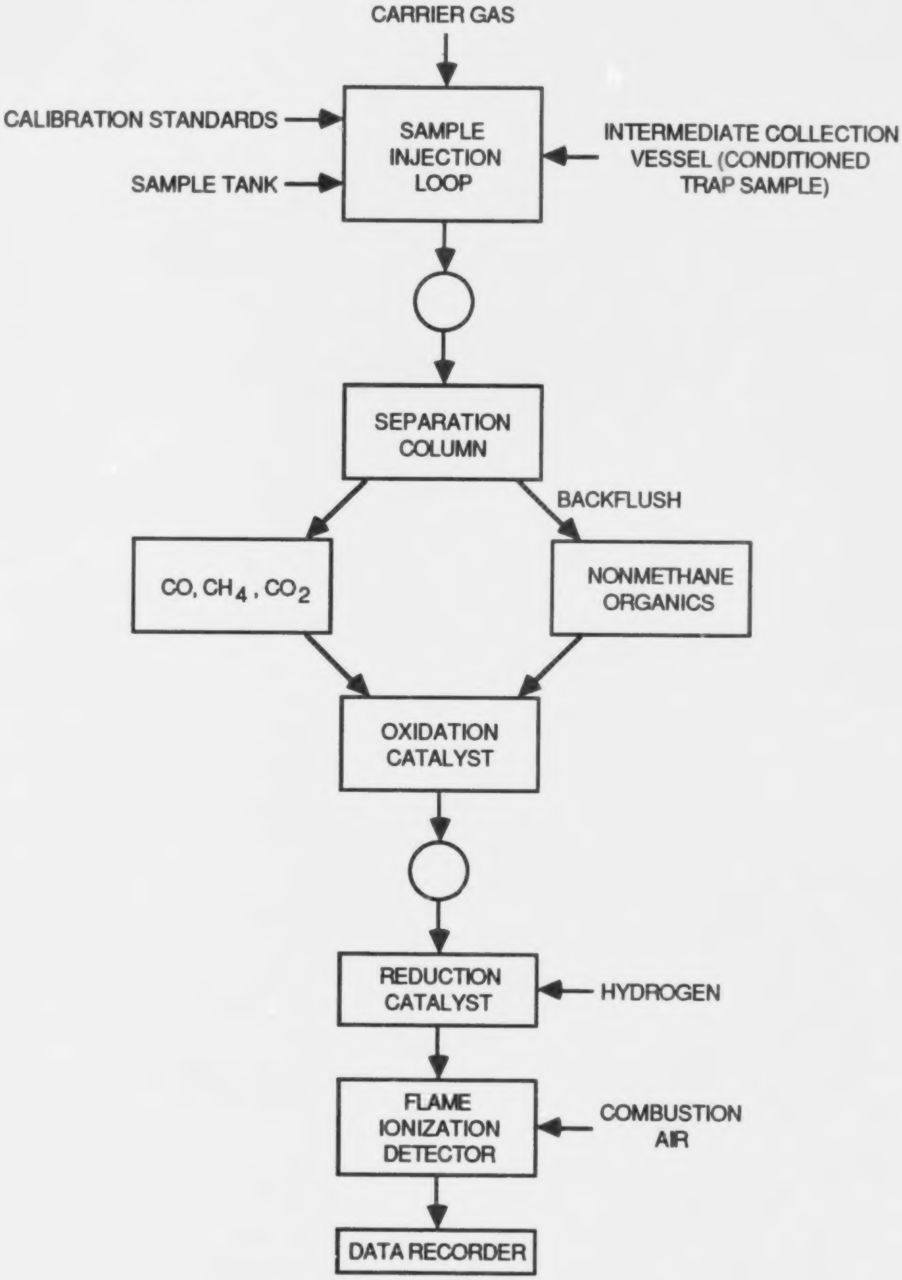
CARRIER GAS
CALIBRATION STANDARDS
SAMPLE TANK
SAMPLE INJECTION LOOP
INTERMEDIATE COLLECTION VESSEL (CONDITIONED TRAP SAMPLE)
SEPARATION COLUMN
BACKFLUSH
CO, CH4, CO2
NONMETHANE ORGANICS
OXIDATION CATALYST
REDUCTION CATALYST
HYDROGEN
FLAME IONIZATION DETECTOR
COMBUSTION AIR
DATA RECORDER

Figure 1.2. Method 25 analytical equipment.

visually check the filter holder for problems, ensure that the unit will properly hold a filter and tighten, and then check the filter holder as part of a unit as described in Subsection 1.1.10. If desired, it may be checked separately by following the checkout procedures in Subsection 1.1.10 that relate to the filter holder.

NOTE: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

1.1.3 *Filter Heating System* - A metal box consisting of an inner and an outer shell separated by insulating material with a heating element in the inner shell capable of maintaining a gas temperature at the filter of 121 ± 3° C (250 ± 5°F).

A suitable heating box is shown in Figure 1.3. The outer shell is a metal box that measures 102 mm x 280 mm x 292 mm (4 in. x 11 in. x 11 1/2 in.), while the inner shell is a metal box measuring 76 mm x 229 mm x 241 mm (3 in. x 9 in. x 9 1/2 in.). The inner box is supported by 13-mm (1/2-in.) phenolic rods. The void space between the boxes is filled with fiberfrax insulation which is sealed in place by means of a silicon rubber bead around the upper sides of the box. A removable lid made in a similar manner, with a 25-mm (1-in.) gap between the parts, is used to cover the heating chamber.

The inner box is heated with a 250-watt cartridge heater, shielded by a stainless steel shroud. The heater is regulated by a thermostatic temperature controller set to maintain a temperature of 121°C (250°F) as measured by a thermocouple in the gas line just before the filter. An additional thermocouple is used to monitor the temperature of the gas behind the filter.

Upon receipt or after construction, visually check the out-of-stack filter box for problems, plug in the heater to ensure operation, check the thermocouple at room temperature to ensure that it is functional, and then check the box as part of the unit as described in Subsection 1.1.10. If desired, it may be checked separately by following the checkout procedures in Subsection 1.1.10 that relate to the filter heating system.

1.1.4 *Condensate Trap* - 9.5-mm (3/8-in.) OD 316 stainless steel tubing bent into a U-shape. Exact dimensions are shown in Figure 1.4. The tubing shall be packed with coarse quartz wool (8 to 15 um), to a density of approximately 0.11 g/cc before bending. While the condensate trap is packed with dry ice in the Dewar, an ice bridge may form between the arms of the condensate trap making it difficult to remove the condensate trap. This problem can be prevented by attaching a steel plate between the arms of the condensate trap in the same plane as the arms to completely fill the intervening space.

Upon receipt or after construction, visually check the condensate trap for problems, ensure proper fittings, ensure proper packing, and then check the condensate trap as part of a unit as described in Subsection 1.1.10. If desired, it may be checked separately by following the checkout procedures in Subsection 1.1.10 that relate to the condensate trap.

1.1.5 *Valve* - Stainless steel shut-off valve for starting and stopping sample flow. Upon receipt, visually check the valve for problems and then check the valve as part of a unit as described in Subsection 1.1.10.

1.1.6 *Metering Valve* - Stainless steel control valve for regulating the sample flow rate through the sampling train. Upon receipt, visually check the valve for problems and then check the valve as part of a unit as described in Subsection 1.1.10.

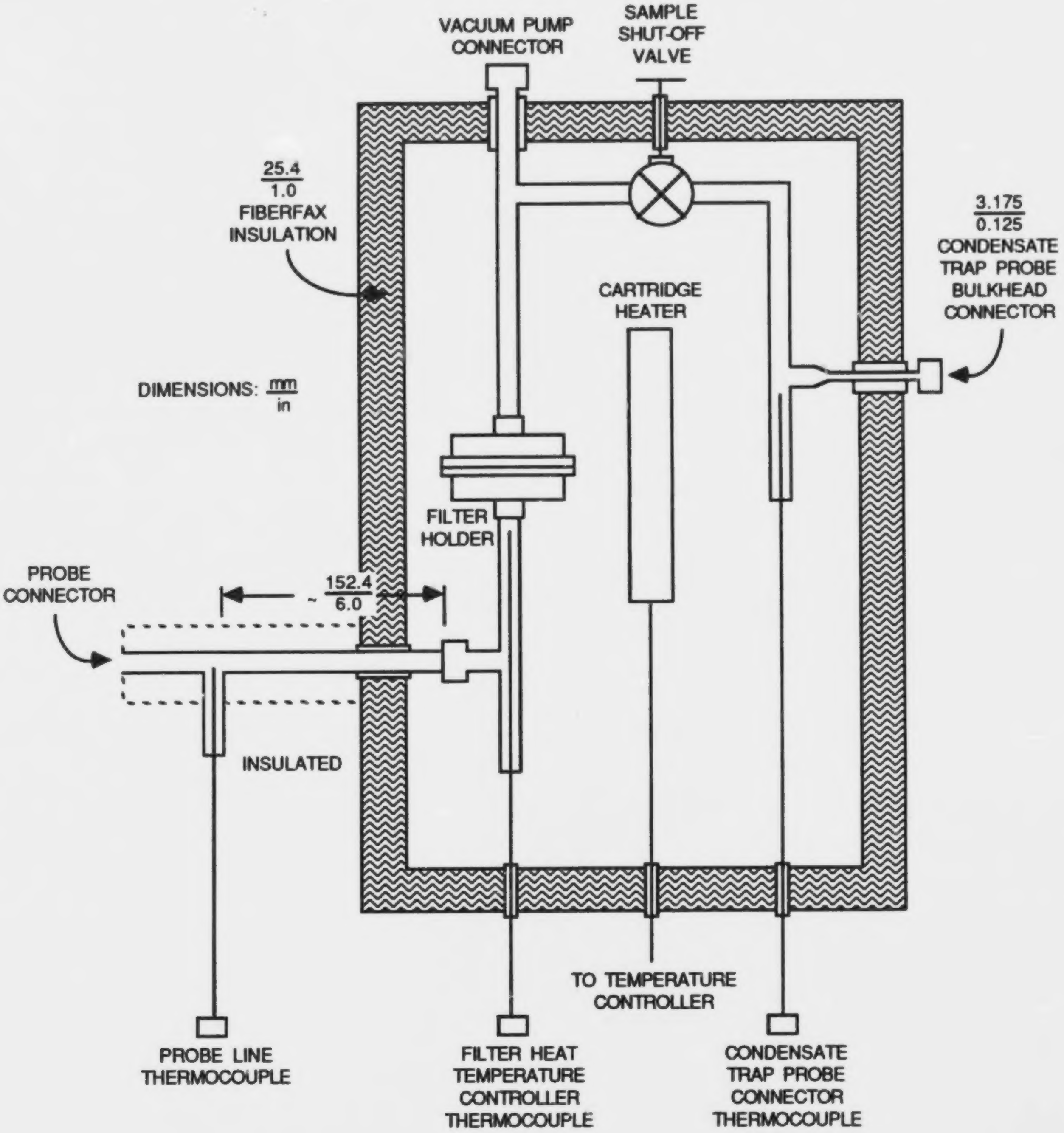
VACUUM PUMP CONNECTOR
SAMPLE SHUT-OFF VALVE
25.4 / 1.0 FIBERFAX INSULATION
3.175 / 0.125 CONDENSATE TRAP PROBE BULKHEAD CONNECTOR
CARTRIDGE HEATER
DIMENSIONS: mm / in
FILTER HOLDER
PROBE CONNECTOR
~ 152.4 / 6.0
INSULATED
TO TEMPERATURE CONTROLLER
PROBE LINE THERMOCOUPLE
FILTER HEAT TEMPERATURE CONTROLLER THERMOCOUPLE
CONDENSATE TRAP PROBE CONNECTOR THERMOCOUPLE

Figure 1.3. Schematic of out-of-stack filter box.

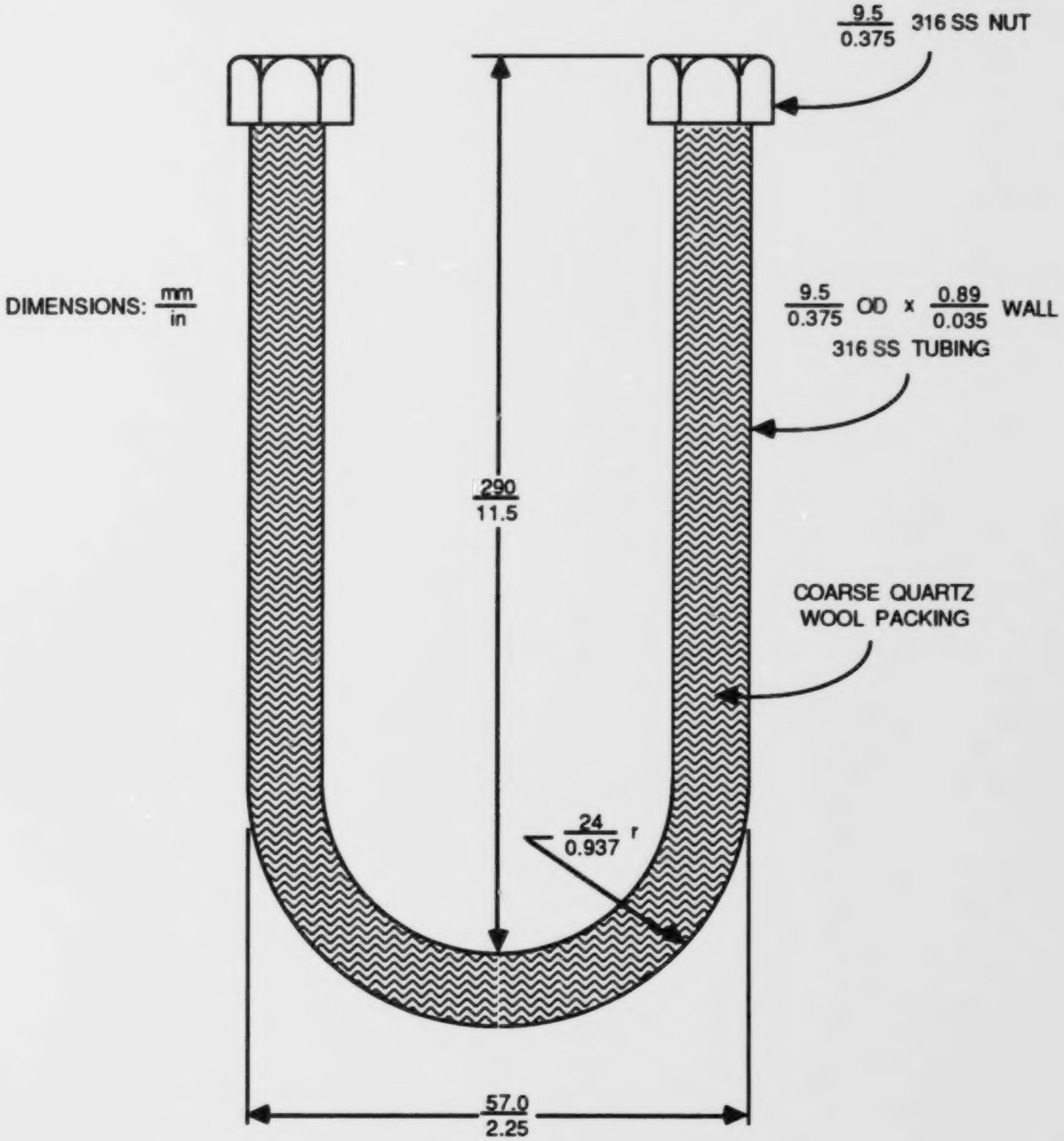
9.5/0.375 316 SS NUT
DIMENSIONS: mm/in
9.5/0.375 OD x 0.89/0.035 WALL
316 SS TUBING
290/11.5
COARSE QUARTZ
WOOL PACKING
24/0.937 r
57.0/2.25

Figure 1.4. Condensate trap.

1.1.7 *Rotameter* - Glass tube with stainless steel fittings, capable of measuring sample flow in the range of 60 to 100 cc/min.

Upon receipt, visually check the rotameter for problems and proper range, ensure proper fittings, and then check it as part of the unit as described in Subsection 1.1.10. If desired the unit may be checked separately by following the checkout procedures in Subsection 1.1.10 that relate to the rotameter.

1.1.8 *Sample Tank* - Stainless steel or aluminum tank with a minimum volume of 4 liters. Upon receipt or after construction, visually check the sample tank for problems and minimum size, ensure proper fittings, and then check the sample tank as part of a unit as described in Subsection 1.1.10. If desired, it may be checked separately by following the checkout procedures in Subsection 1.1.10 that relate to the sample tank.

1.1.9 *Mercury Manometer or Absolute Pressure Gauge* - Capable of measuring pressure to within 1 mm Hg in the range of 0 to 1,200 mm Hg. Upon receipt or after construction, visually check the pressure gauge for problems, ensure proper fittings, proper range, and proper sensitivity, and then check it as part of a unit as described in Subsection 1.1.10. If desired, it may be checked separately by following the checkout procedures in Subsection 1.1.10 that relate to the pressure gauge.

1.1.10 *Vacuum Pump* - Capable of evacuating a container to an absolute pressure of 10 mm Hg. Upon receipt, visually check the vacuum pump for problems. Ensure proper fittings, attach the pump to a vacuum gauge, and determine if the pump is capable of evacuating to an absolute pressure of 10 mm Hg. Check it as part of a unit as described below. If desired, it may be checked separately by following the checkout procedures below that relate to the pump.

1. Calibrate all thermocouples as described in Section 3.17.2. If the thermocouples are not within 3°C (5°F) of the true temperature, reject or repair and recalibrate.
2. Calibrate the rotameters as described in Section 3.17.2. If the rotameters cannot determine the flowrate to within 10 percent of the actual flowrate over the indicated range, reject or repair and then recalibrate.
3. Calibrate and leak check the sample tank as described in Section 3.17.2. The tank is acceptable if no change in tank vacuum is noted over a 1 hour period.
4. With the sample tank evacuated, assemble the sampling train (including placing a filter in the filter holder) as shown in Figure 1.1, with the exception that is not necessary to use dry ice to cool the condensate trap. Plug the probe tip and verify that the tank valve is closed. Turn on the vacuum pump, and evacuate the sampling system from the probe tip to the sample tank valve to an absolute pressure of 10 mm Hg or less. Close the purge valve, turn off the pump, wait a period of 5 minutes, and recheck the indicated vacuum. The method allows a leak rate of 1 percent of the sampling rate, but for this initial check of new equipment it is recommended that the criteria be increased to no noticeable leak. If a leak is noted, find the source of it and reject, repair, or replace the component(s) and repeat the leak check until satisfactory results are obtained.
5. Release the vacuum and then unplug the probe tip. Set the probe temperature controller to 129°C (265°F) and the filter temperature

controller to 121°C (250°F). Allow the probe and filter to heat for about 30 minutes. Close the sample valve, open the purge valve, start the vacuum pump, and set the flow rate between 60 and 100 cc/min. If the purge system draws gas through the probe and filter at the desired rate, the purge system is acceptable. If the purge system does not provide the desired flow rate, repair or replace the system or problem component and repeat the check.

6. When the temperature at the exit ends of the probe and filter are within the specified range, close the purge valve and stop the pump. Open the sample valve and the sample tank valve. Using the flow control valve, set the flow through the sample train to the maximum rate that would be normally used (i.e., 100 cc/min). Operate the system for about 30 minutes, adjusting the flow rate as necessary to maintain a constant rate (±10 percent). The temperature of the probe and filter must remain in the specified range and the flowrate should be adjustable. If the flow rate and temperatures can be maintained in the proper range, conduct another leak check as described above in Step 4. If the flow rate and/or temperatures cannot be maintained in the proper range, repair or replace system or problem component(s) and repeat sampling check and leak checks.

1.2 Analysis

The analysis equipment consists primarily of an organic condensate recovery system for processing sample traps and a nonmethane organic (NMO) analyzer for analysis of CO_2 and NMO. The recovery system can be constructed from commercially available components and components fabricated in a machine shop. The NMO analyzer can be purchased as a commercial unit or assembled in the laboratory by modification of a packed column gas chromatograph.

1.2.1 *Condensate Recovery Apparatus* - The system for the recovery of the organics captured in the condensate trap consists of a heat source, oxidation catalyst, nondispersive infrared (NDIR) analyzer, and an intermediate collection vessel (ICV). Figure 1.5 is a schematic of a typical system. The system shall be capable of proper oxidation and recovery, as specified in Section 3.17.5. The following major components are required:

Heat Source - Sufficient to heat the condensate trap (including connecting tubing) to a temperature of 200°C. A system using both a heat gun and an electric tube furnace is recommended. Upon receipt, visually check the heat gun and/or electric tube furnace for any defects. Check any device for heating before use to determine if a condensate trap temperature of 200°C can be achieved.

Heat Tape - Sufficient to heat the connecting tubing between the water trap and the oxidation catalyst to 100°C. Upon receipt, visually check the heating tape for any defects and test to ensure heating of the connecting tubing to 100°C.

Oxidation Catalyst - A suitable length of 9.5-mm (3/8-in.) OD Inconel 600 tubing packed with 15 cm (6 in.) of 3.2-mm (1/8-in.) diameter 19 percent chromia on alumina pellets. The catalyst material is packed in the center of the catalyst tube with quartz wool packed on either end to hold it in place. The catalyst tube shall be mounted vertically in a 650°C tube furnace. After construction, visually check the catalyst tube for problems and test the tube heater to ensure heating to 650°C.

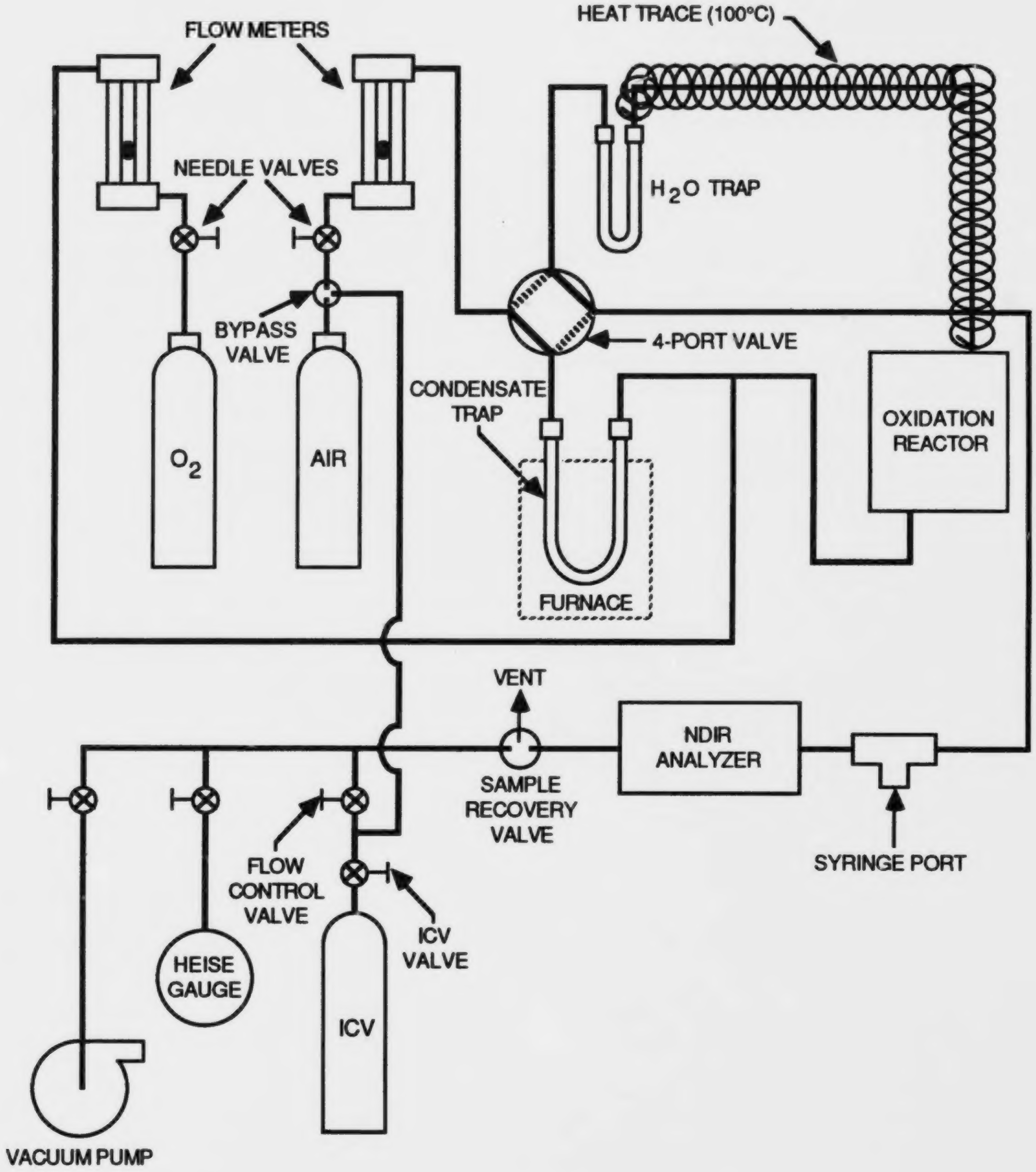
FLOW METERS
HEAT TRACE (100°C)
NEEDLE VALVES
H_2O TRAP
BYPASS VALVE
4-PORT VALVE
CONDENSATE TRAP
O_2
AIR
OXIDATION REACTOR
FURNACE
VENT
NDIR ANALYZER
SAMPLE RECOVERY VALVE
SYRINGE PORT
FLOW CONTROL VALVE
ICV VALVE
HEISE GAUGE
ICV
VACUUM PUMP

Figure 1.5. Condensate recovery system.

Water Trap - Leak proof, capable of removing moisture from the gas stream. Upon receipt or after construction, visually check the water trap for defects, especially leakage problems.

Syringe Port - A 6.4-mm (1/4-in.) OD stainless steel tee fitting with a rubber septum placed in the side arm. After construction, visually check the syringe port for defects, especially leakage around the rubber septum.

NDIR Detector - Capable of indicating CO_2 concentration in the range of 0 to 5 percent, to monitor the progress of combustion of the organic compounds from the condensate trap. Upon receipt, visually check the NDIR detector for defects. Zero and span the analyzer according to the manufacturer's directions.

Flow-Control Valve - Stainless steel, to maintain the trap conditioning system near atmospheric pressure. Upon receipt, visually check the flow control valve for defects.

Intermediate Collection Vessel (ICV) - Stainless steel or aluminum, equipped with a female quick connect. Tanks with nominal volumes of at least 6 liters are recommended. Upon receipt, visually check the ICV for defects.

Determine the ICV volume by weighing it while empty and then filling it with deionized distilled water; weigh to the nearest 5 g and record the difference between the full and empty weights as the tank volume in ml. Alternatively, measure the volume of water used to fill the tank to the nearest 5 ml.

An alternative to using the rigid containers is the use of flexible bags made of Tedlar or Teflon film. However, the calculations given in Section 3.17.6 are no longer appropriate since the gas volumes must be measured directly. It is the responsibility of the tester to apply calculations which are consistant with directly measured gas volumes.

Mercury Manometer or Absolute Pressure Gauge - Capable of measuring pressure to within 1 mm Hg in the range of 0 to 1,200 mm Hg. Upon receipt, visually check the manometer or pressure gauge for defects and proper operating range and precision.

Syringe - 10-ml gas-tight, glass syringe equipped with an appropriate needle. Upon receipt, visually check the syringe for defects and proper volume.

1.2.2 *NMO Analyzer* - The NMO analyzer is a gas chromatograph (GC) with backflush capability for NMO and CO_2 analysis and is equipped with an oxidation catalyst, reduction catalyst, and FID. Figures 1.6 and 1.7 are schematics of a typical NMO analyzer. This semicontinuous GC/FID analyzer shall be capable of: (1) separating CO, CO_2, and CH_4 from NMO; (2) reducing the CO_2 to CH_4, and quantifying as CH_4; and (3) oxidizing the NMO to CO_2, reducing the CO_2 to CH_4 and quantifying as CH_4 according to Section 3.17.5. The NMO analyzer consists of the following major components:

Oxidation Catalyst - A suitable length of 9.5-mm (3/8-in.) OD Inconel 600 tubing packed with 5.1 cm (2 in.) of 19 percent chromia on 3.2-mm (1/8-in.) alumina pellets. The catalyst material is packed in the center of the tube and supported on either side by quartz wool. The catalyst tube must be mounted vertically in a 650°C furnace. After construction, visually inspect the oxidation catalyst for defects and ensure that the tube furnace is capable of heating to 650°C.

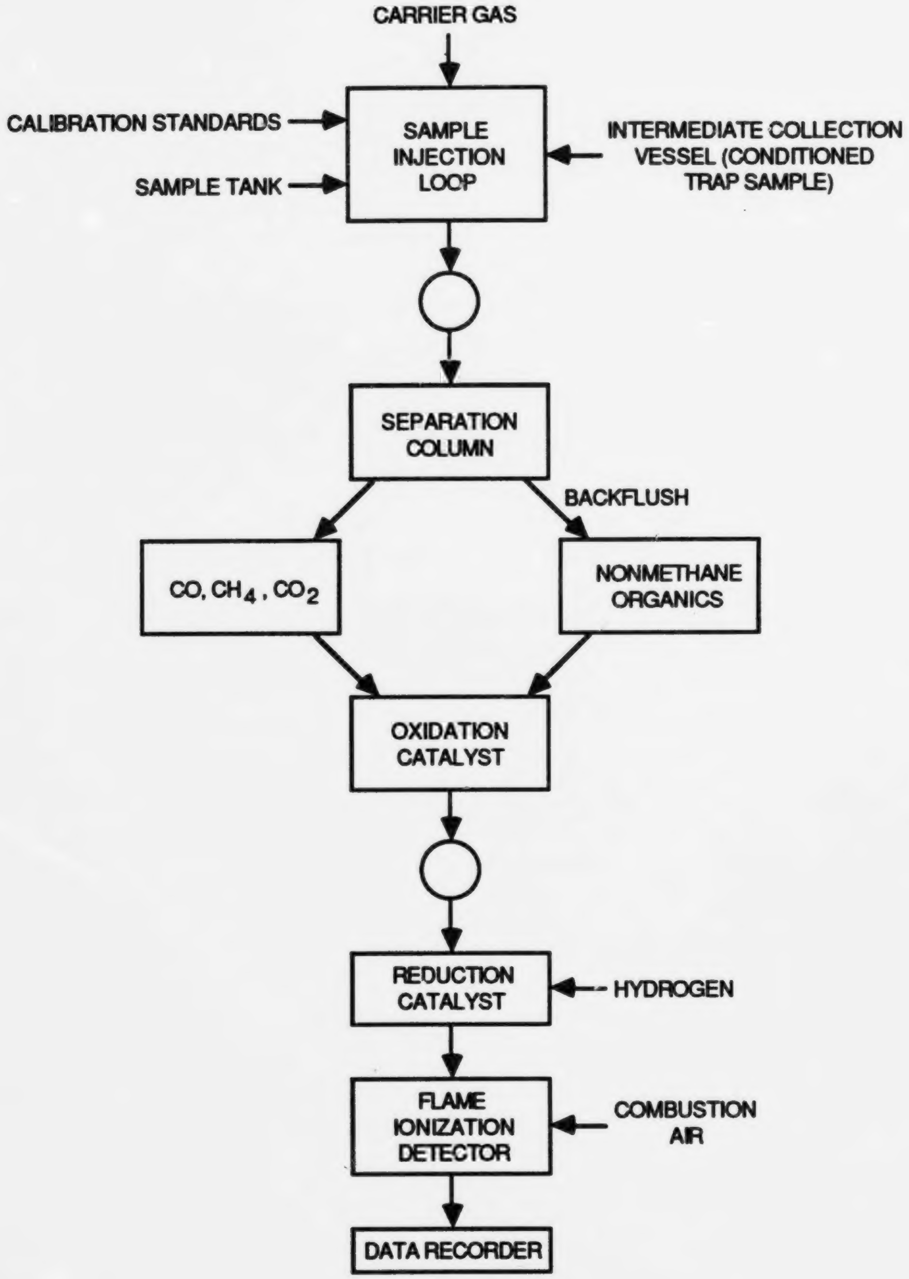
CARRIER GAS
CALIBRATION STANDARDS
SAMPLE TANK
SAMPLE INJECTION LOOP
INTERMEDIATE COLLECTION VESSEL (CONDITIONED TRAP SAMPLE)
SEPARATION COLUMN
BACKFLUSH
CO, CH4, CO2
NONMETHANE ORGANICS
OXIDATION CATALYST
REDUCTION CATALYST
HYDROGEN
FLAME IONIZATION DETECTOR
COMBUSTION AIR
DATA RECORDER

Figure 1.6. Simplified schematic of nonmethane organic (NMO) analyzer.

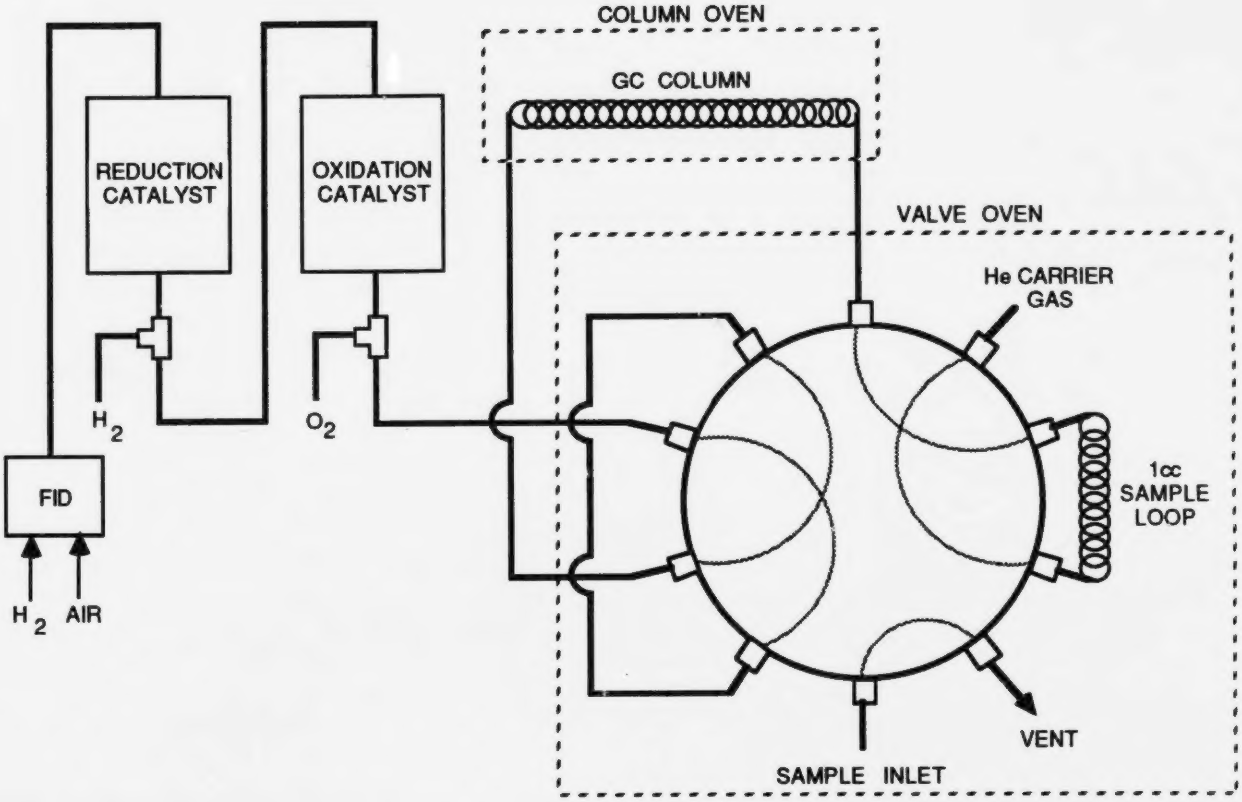
COLUMN OVEN
GC COLUMN
REDUCTION CATALYST
OXIDATION CATALYST
VALVE OVEN
He CARRIER GAS
H_2
O_2
FID
1cc SAMPLE LOOP
H_2
AIR
VENT
SAMPLE INLET

Figure 1.7. Nonmethane organic (NMO) analyzer.

Reduction Catalyst - A 7.6-cm (3-in.) length of 6.4-mm (1/4-in.) OD Inconel tubing fully packed with 100-mesh pure nickel powder. The catalyst tube must be mounted vertically in a 400°C furnace. After construction, visually inspect the reduction catalyst for defects and ensure that the tube furnace is capable of heating to 400°C.

Separation Column(s) - A 30-cm (1-ft) length of 3.2-mm (1/8-in.) OD stainless steel tubing packed with 60/80 mesh Unibeads is followed by a 61-cm (2-ft) length of 3.2-mm (1/8-in.) OD stainless steel tubing packed with 60/80 mesh Carbosieve G. The Carbosieve and Unibeads columns must be baked separately at 200°C with carrier gas flowing through them for 24 hours before initial use. The columns should then be connected to each other with a 1/8-inch stainless steel union. The column series should be connected to the sample injection valve so that the sample loop contents will be injected onto the head of the Unibeads 1S column.

Sample Injection System - A 10-port GC sample injection valve fitted with a sample loop properly sized to interface with the NMO analyzer (1-cc loop recommended). Upon receipt, visually inspect the sample injection system for defects and check for proper number of ports and valve fitting size for the connecting tubing used (1/16- or 1/8-in.).

FID - An FID meeting the following specifications is required:

1. Linearity - A linear response (± 5 percent) over the operating range as demonstrated by the procedures established in Section 3.17.5.
2. Range - A full scale range of 10 to 50,000 ppm CH_4. Signal attenuators shall be available to produce a minimum signal response of 10 percent of full scale.

Data Recording System - Analog strip chart recorder or digital integration system compatible with the FID for permanently recording the analytical results. Upon receipt, visually inspect the data recording system for defects and test according to manufacturer's instructions.

1.2.3 *Other Analysis Apparatus* -

Barometer - Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 1 mm Hg. Upon receipt, visually check the barometer for defects.

Thermometer - Capable of measuring the laboratory temperature to within 1°C. Upon receipt, visually check the thermometer for defects.

Vacuum Pump - Capable of evacuating to an absolute pressure of 10 mm Hg or less. Upon receipt, visually check the vacuum pump for defects and test to ensure capability to reach proper vacuum

Syringes - 10 ul and 50 ul liquid injection syringes. Upon receipt, visually check syringes for defects and proper volume.

Liquid Sample Injection Unit - 316 stainless steel U-tube constructed as shown in Figure 1.8 for performing condensible organic recovery efficiency tests. After construction, visually check the unit for problems, especially leakage around the rubber septum.

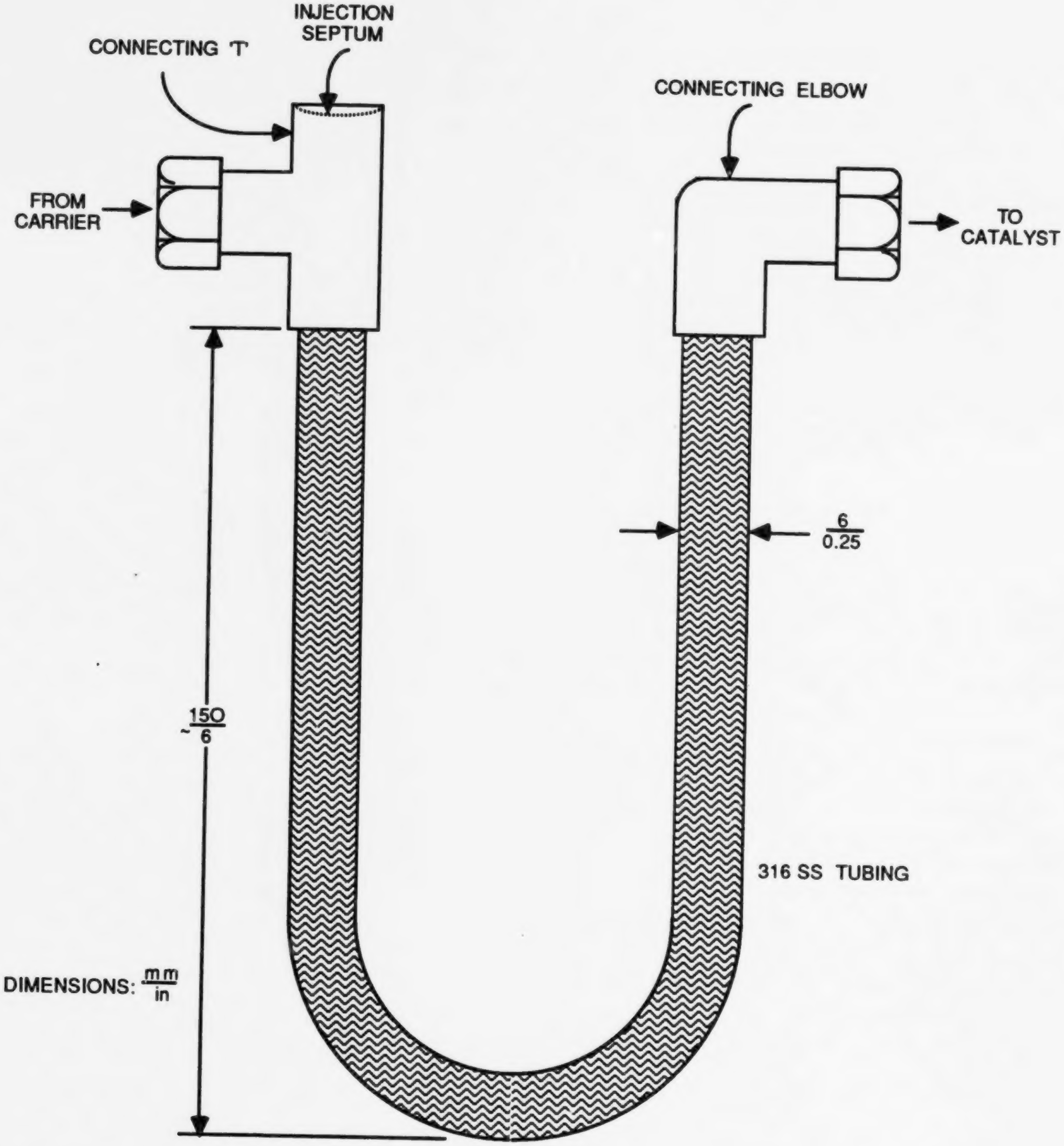
INJECTION SEPTUM
CONNECTING 'T'
CONNECTING ELBOW
FROM CARRIER
TO CATALYST
$\frac{6}{0.25}$
$\sim\frac{150}{6}$
316 SS TUBING
DIMENSIONS: $\frac{mm}{in}$

Figure 1.8. Liquid sample injection unit.

1.3 Reagents and Other Supplies

Unless otherwise indicated, all reagents should meet the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS); otherwise, use the best available grade.

1.3.1 *Sampling* - The following are required for sampling:

Crushed Dry Ice - Crushed dry ice is needed to cool the condensate (U-tube) trap during sampling for better collection of organics and to keep it cold until analysis. There are no specifications on the dry ice.

Coarse Quartz Wool - Coarse quartz wool, 8 to 15 um in size, is needed to pack the condensate (U-tube) traps in the laboratory for sampling. The packing should not be conducted in the field prior to testing since a trap packed with new quartz wool must be taken to 300°C and then blank checked prior to its use in a field test. Upon receipt, check the specifications of the quartz wool. If the proper wool has been sent, it should be acceptable. If the specifications are not met, reorder the proper item.

Filters - Glass fiber filters, without organic binder are needed to remove organic particulate matter from the gas stream during sample collection. Typically, filters used for Method 5 tests will be satisfactory, if no organic binders are present. If organic binders are present, they may be released during testing and positively bias the results. If the tester is not certain about the presence of organic binders in a glass fiber filter, it should be placed in a furnace at 300°C for 2 hours which will remove any organic binders present. This procedure, however, may make the filter more brittle resulting in a greater need for caution in handling. A check on the amount of organic binder lost can be determined by weighing the filter both before and after heating. If a significant weight loss occurs (1 mg per filter), the filters probably contain organic binders. They may still be used, but it is recommended that another type of filter be ordered and checked in the same manner, since removing the binders with heat may make the filters too brittle to use safely.

1.3.2 *NMO Analysis* - Several gases are needed for NMO analysis depending on the exact analyzer used for analysis. It is critical that all gases meet the requirements for background contamination, to ensure that a low background level is present during sample analysis. The following gases are needed for NMO analysis:

Carrier Gases - Depending on the exact NMO analyzer, two carrier gases will be needed for analysis. Typically zero grade helium (He) and zero grade oxygen (O_2) containing less than 1 ppm CO_2 and less than 0.1 ppm C as hydrocarbon will be required. Upon receipt, check the label for manufacturer's specifications. If the gases do not meet the above specifications, they should be returned to the supplier, and new gases obtained and checked.

Fuel Gas - Typically zero grade hydrogen (H_2) cylinder gas is needed as a fuel gas. The hydrogen should be 99.999 percent pure. Upon receipt, check the label and manufacturer's specifications. If the gas does not meet these specifications, return it to the supplier, and obtain and check a new cylinder.

Combustion Gas - Zero grade air or O_2 (as required by the GC detector) is needed. Upon receipt, check the specifications. If the gas does meet the required specifications, return it to the supplier and obtain new gas and recheck.

1.3.3 *Condensate Recovery* - Two gases are needed for condensate recovery. It is critical that all gases meet the requirements for background contamination, to ensure that a low background level is present during sample analysis. The following gases are needed for condensate recovery:

Carrier Gas - Zero grade air, containing less than 1 ppm C as hydrocarbons is needed as a carrier for purging the CO_2 from the trap into the sample tank and then purging the sample from the trap during the oxidation step into an intermediate collection vessel. Upon receipt, the manufacturer's specifications should be checked and the gases analyzed for background levels as described in Section 3.17.5. If the gas does not meet the requirements, it should be replaced and the new gas checked.

Auxiliary Oxidant - Zero grade O_2, containing less than 1 ppm C as hydrocarbons may be needed during the oxidation of the condensate trap sample. Upon receipt, the gas should be checked as described above. Zero grade air may be used instead of zero grade O_2 if the condensible organic recovery efficiency test (described in Section 3.17.5) can be passed.

1.3.4 *Condensate Recovery Performance* - The following liquid reagents are needed:

Hexane - ACS grade hexane is needed for liquid injection into the liquid sample injection unit of the condensate recovery system to conduct the condensible organic recovery efficiency test. Upon receipt, check the container of hexane to ensure that the ACS grade specifications are met. If they are not met, return it to the supplier, obtain a new container, and recheck.

Decane - ACS grade decane is needed for liquid injection into the liquid sample injection unit of the condensate recovery system to conduct the condensible organic recovery efficiency test. Upon receipt, check the container of decane to ensure that the ACS grade specifications are met. If they are not met, return it to the supplier, obtain a new container, and recheck.

1.3.5 *Calibration Gases for Analysis* - The concentrations of all calibration gases should be traceable to National Institute for Standards and Technology (NIST) Standards. For those calibration gases that have corresponding gaseous NIST standards (i.e., propane and carbon dioxide), traceability should be established via the EPA's Revised Traceability Protocol No. 1 (Reference 9). For the remaining calibration gases, traceability should be established to gravimetric NIST standards. Traceability to NIST is necessary because some calibration gases with certificates of analysis have shown significant errors when they were compared with NIST standards. Specialty gas manufacturers should certify the accuracy of their calibration gases.

Revised Traceability Protocol No. 1 compares the concentrations of calibration gases to those of gaseous NIST Standard Reference Materials (SRMs) or to those of gaseous NIST/EPA Certified Reference Materials (CRMs), which are accepted as equivalent to SRMs (Reference 10). Although explicit accuracy specifications for these EPA protocol gases do not exist, accuracy assessments by EPA have found that many EPA protocol gases are accurate to within 2 percent and that most are accurate to within 5 percent (Reference 11). EPA protocol gases may be purchased from most specialty gas manufacturers.

For all calibration gases, the manufacturer must recommend a maximum shelf life (i.e., the length of time during which the gas concentration is not expected to change by more than 5 percent from its certified value). EPA protocol gases have a certification period of 18 months, after which they should be recertified. Specialty gas manufacturers should be able to produce stability data to support the maximum shelf life recommendation. The data should be for the same compound, balance gas, and approximate concentration as requested.

Do not store the calibration gas cylinders in areas subject to extreme temperature changes. Before each calibration, check the pressure of the calibration gas in the cylinder and replace any cylinders with a pressure less than 1500 kilopascals (or 200 lb/sq. in.).

The following calibration gases are required.

Oxidation Catalyst Efficiency Check Calibration Gas - A calibration gas with a nominal concentration of 1 percent methane in air is required for the oxidation catalyst efficiency check. Upon receipt of the calibration gas, check its certificate of analysis to ensure that the correct concentration has been sent. Verify its certified concentration against calibration gases currently being used for analysis. Be certain that balance gas differences do not cause measurement errors in the analyzer used for verification. The verified concentration should agree within 2 percent of the certified concentration. If 2 percent agreement is obtained, the certified concentration can be used. If the agreement is between 2 and 5 percent, the verified concentration can be used. If the agreement is greater than 5 percent, first inspect the analyzer for malfunction and reverify the calibration gas. If the reverified agreement is also greater than 5 percent, consult with the specialty gas manufacturer about replacement of the calibration gas.

The organic condensate recovery system oxidation catalyst efficiency check compares the concentrations of the methane and CO_2 calibration gases. The concentrations of the two calibration gases must agree within 2 percent. Be certain to compare the calibration gases on a ppm carbon basis. If such agreement is not obtained, the problem may lie in the catalyst or in the calibration gases. Inspect or replace the catalyst and reverify the calibration gases' concentrations before repeating the check. Consult with the specialty gas manufacturer about replacement of the calibration gases if the lack of agreement persists.

NMO Response Linearity and Calibration Gases - Three calibration gases with nominal concentrations of 20, 200, and 3,000 ppm propane in air are required for the NMO linearity check and to determine the calibration response factor. Upon receipt of the calibration gases, check their certificates of analysis to ensure that the correct concentrations have been sent. Verify the certified concentrations against SRMs using revised Traceability Protocol No. 1 or against calibration gases currently being used for analysis as described in Section 3.17.5. Be certain that balance gas differences do not cause measurement errors in the analyzer used for certification. The verified concentration should agree within 2 percent of the certified concentration. If 2 percent agreement is obtained, the certified concentration can be used. If the agreement is greater than 5 percent, first inspect the analyzer for malfunction and reverify the calibration gas. If the reverified agreement is also greater than 5 percent, consult with the specialty gas manufacturer about replacement of the calibration gas.

CO_2 Response Linearity and Calibration Gases - Three calibration gases with nominal concentrations of 50 ppm, 500 ppm, and 1 percent carbon dioxide in air are required to determine the overall mean CO_2 response factor. Upon receipt of the calibration gases, check their certificates of analysis to ensure that the correct concentrations have been sent. Verify the certified concentrations against SRMs using revised Traceability Protocol No. 1 or against calibration gases currently being used for analysis as described in Section 3.17.5. Be certain that balance gas differences do not cause measurement errors in the analyzer used for certification. For example, carbon dioxide in air response factors will differ from carbon dioxide in nitrogen response factors for nondispersive infrared analyzers due to pressure broadening effects. The verified concentration should agree within 2 percent of the certified concentration. If 2 percent agreement is obtained, the certified concentration can be used. If the agreement is between 2 and 5 percent, the verified concentration can be used. If the agreement is greater than 5 percent, first inspect the analyzer for malfunction and reverify the calibration gas. If the reverified agreement is also greater than 5 percent, consult with the specialty gas manufacturer about replacement of the calibration gas.

The analyzer linearity check and NMO calibration requires 10 percent agreement between the propane and CO_2 calibration gases. Be sure to compare the calibration gases on a ppm carbon basis. If such agreement cannot be obtained, inspect the analyzer for malfunction and reverify the calibration gases. Consult with the specialty gas manufacturer about replacement of the calibration gases if the lack of agreement persists.

NMO Analyzer System Performance Check Calibration Gases - The four following calibration gases are needed for the NMO analyzer system check:

1. Propane Mixture - A calibration gas with nominal concentrations of 50 ppm carbon monoxide, 50 ppm methane, 2 percent carbon dioxide, and 20 ppm propane in air is required for the NMO analyzer system performance check. Upon receipt of the calibration gas, check its certificate of analysis to ensure that the correct concentrations have been sent. Replace the calibration gas if the purchase specifications have not been met. If the specifications have been met, conduct the NMO calibration check. The response factor for this calibration gas should be within 5 percent of the overall mean response factor for the propane in air calibration gases. If such agreement cannot be obtained, inspect the equipment for malfunction and verify the concentrations of the components in the calibration gas. Consult with the specialty gas manufacturer about replacement of the calibration gas if the lack of agreement persists.
2. Hexane Calibration Gas - A calibration gas with a nominal concentration of 50 ppm hexane in air is required for the NMO analyzer system performance check. Upon receipt of the calibration gas, inspect its certificate of analysis to ensure that the correct concentration has been sent. Replace the calibration gas if the purchase specifications have not been met. If the specifications have been met, conduct the NMO calibration check. The response factor for this calibration gas should be within 5 percent of the overall mean response factor for the propane in air calibration gases. Be sure to compare the calibration gases on a ppm carbon basis. If such agreement cannot be obtained, inspect the equipment for malfunction and, if possible, verify the concentration of the calibration gas with an analyzer employing a different analytical principle (e.g., gas chromatography). Consult with the specialty gas manufacturer about replacement of the

calibration gas if the lack of agreement persists.

3. Toluene Calibration Gas - A calibration gas with a nominal concentration of 20 ppm toluene in air is required for the NMO analyzer system performance check. Upon receipt of the calibration gas, check its certificate of analysis to ensure that the correct concentration has been sent. Replace the calibration gas if the purchase specifications have not been met. If the specifications have been met, conduct the NMO calibration check. The response factor for this calibration gas should be within 5 percent of the overall mean response factor for the propane in air calibration gases. Be sure to compare the calibration gases on a ppm carbon basis. If such agreement cannot be obtained, inspect the equipment for malfunction and, if possible, verify the concentration of the calibration gas with an analyzer employing a different analytical principle (e.g., gas chromatography). Consult with the specialty gas manufacturer about replacement of the calibration gas if the lack of agreement persists.
4. Methanol Calibration Gas - A calibration gas with a nominal concentration of 100 ppm methanol in air is required for the NMO analyzer system performance check. Upon receipt of the calibration gas, check its certificate of analysis to ensure that the correct concentration has been sent. Replace the calibration gas if the purchase specifications have not been met. If the specifications have been met, conduct the NMO calibration check. The response factor for this calibration gas should be within 5 percent of the overall mean response factor for the propane in air calibration gases. Be sure to compare the calibration gases on a ppm carbon basis. If such agreement cannot be obtained, inspect the equipment for malfunction and, if possible, verify the concentration of the calibration with an analyzer employing a different analytical principle (e;g., gas chromatography). Consult the specialty gas manufacturer about replacement of the calibration gas if the lack of agreement persists.
   Note: Little is currently known about the stability of calibration gases containing methanol in air. Special attention should be given to the stability of this calibration gas.

Table 1.1. ACTIVITY MATRIX FOR PROCUREMENT OF APPARATUS AND SUPPLIES

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Sampling			
Heated probe	Stainless steel capable of heating to 129°C (365°F) at exit end	Upon receipt, check heater and thermometer with system	Repair or replace, and recheck
Filter holder	Stainless steel with inside diameter of 25 mm (15/16 in.) and Teflon O-ring	Check unit to ensure that filter is held properly	Repair or replace, and recheck
Filter heating system	Metal box consisting of an inner and an outer shell separated by fiber fran insulation with a heater capable of maintaining a gas temperature of 121°C ±3°C (250 ±5°F)	Visually check, ensure heater is operational, check thermocouple at room temperature, check box as part of the unit (Subsection 1.1.10)	Repair or replace and recheck
Condensate trap	9.5 mm (3/8 in.) OD 316 stainless steel tubing U-tube shaped, packed with coarse quartz wool	Visually check, ensure proper fittings, proper packing, check as part of unit	Repair or replace and recheck
Valve	Stainless steel shut-off valve	Visually check valve, check as part of unit	Repair or replace and recheck
Metering valve	Stainless steel control valve	Visually check valve, check as part of unit	Repair or replace and recheck
Rotameter	Glass tube with stainless steel fittings, capable of measuring sample flow of 60 to 100 cc/min	Visually check, ensure proper range and proper fittings, check as part of unit	Reject or repair, then recalibrate

(Continued)

Table 1.1 (Continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Sample tank	Stainless steel or aluminum tank with a minimum volume of 4 liters	Visually check, ensure proper fittings, minimum size, check as part of unit	Repair or replace and recheck
Mercury manometer or absolute pressure gauge	Capable of measuring pressure to within 1 mm Hg. in the range of 0 to 1,200 mm Hg	Visually check, ensure proper fittings, range, sensitivity, check as part of unit	Determine correction factor or reject
Vacuum pump	Capable of evacuating to an absolute pressure of 10 mm Hg	Visually check, ensure proper fittings, determine for evacuating 10 mm Hg, check as part of unit	Repair or replace and recheck
Analysis			
Condensate recovery apparatus			
1. Heat source	Sufficient to heat condensate trap to 200°C	Visually check, conduct heat check	Repair or replace and recheck
2. Heat tape	Sufficient to heat connecting tubing to 100°C	Visually check, conduct heat check	Replace and recheck
3. Oxidation catalyst	9.5 mm (3/8 in.) OD Inconel 600 tubing packed with 15 cm (6 in.) of 3.2 cm (1/8 in.) diameter 19 percent chromia or alumina pellets	Visually check	Repair or replace and recheck
4. Water trap	Leak proof, capable of removing moisture from gas stream	Visually check, conduct leak check	Repair or replace and recheck
5. Syringe port	6.4 mm (1/4 in.) OD stainless steel	Visually check	Repair or replace and recheck

(Continued)

Table 1.1 (Continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
6. NDIR detector	Capable of indicating CO_2 concentration in the range of 0 to 5 percent		Repair or replace and recheck
7. Flow-control valve	Stainless steel	Visually check, check pressure rating	Repair or replace and recheck
8. Intermediate collection vessel	Stainless steel or aluminum, nominal volume of 6 liters	Visually check, ensure proper volume	Repair or replace and recheck
9. Mercury manometer or absolute pressure gauge	Capable of measuring pressure to within 1 mm Hg in the range of 0 to 1,200 mm Hg	Visually check, ensure ensure proper sensitivity	Determine correction factor or reject
10. Syringe	10 ml gas-tight, glass	Visually check	Repair or replace and recheck
NMO Analyzer			
1. Oxidation catalyst	Suitable length of 9.5 mm OD Inconel 600 tubing packed with 5.1 cm of 19 percent chromia on 3.2 mm alumina pellets	Visually check	Replace and recheck
2. Reduction catalyst	7.6 cm length of 6.4 mm OD Inconel tubing packed with 100 mesh pure nickel powder	Visually check	Replace and recheck

(Continued)

Table 1.1 (Continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
3. Separation column	30 cm length of 3.2 mm OD stainless steel tubing packed with 60/80 mesh Unibeads 1S followed by 61 cm length of 3.2 mm OD stainless steel tubing packed with 60/80 mesh Carbosieve G	Visually check	Replace and recheck
4. Sample injection system	10-port GC sample injection valve fitted with a sample loop	Visually check, sample loop should be of proper size to interface with NMO analyzer	Repair or replace and recheck
5. FID	A linear response of ± 5 percent over operating range of 10 to 50,000 ppm CH_4, minimum signal response of 10 percent of full scale	Upon receipt use procedure established in Section 3.17.5	Return to manufacturer or repair and recheck
6. Data recording system	Analog strip chart compatible with FID	Upon receipt check as recommended by manufacturer	Repair or return to manufacturer
Reagents and other supplies			
Sampling			
1. Crushed dry ice	No specifications	None	None
2. Coarse quartz wool	8 to 15 um in size	Upon receipt check manufacturer's specifications	Return to manufacturer
3. Filters	Glass fiber filters without organic binders	Check for presence of organic binders	Return to manufacturer

(Continued)

Table 1.1 (Continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
NMO analysis			
1. Carrier gas	As specified by owner's manual, less than 1 ppm CO_2 and 0.1 ppm C as hydrocarbon	Upon receipt check label and manufacturer's specifications	Return to supplier and check new gas
2. Fuel gas	As specified by owner's manual	Upon receipt check label and manufacturer's specifications	Return to supplier and check new gas
3. Combustion gas	As specified by owner's manual	Upon receipt check label and manufacturer's specifications	Return to supplier and check new gas
Condensate analysis			
1. Carrier gas	Zero grade air containing less than 1 ppm C as hydrocarbon	Same as above	Same as above
2. Auxillary O_2	Zero grade O_2 containing less than 1 ppm C as hydrocarbon	Same as above	Same as above
3. Hexane	ACS grade hexane	Visually check to ensure ACS grade	Return to manufacturer and check new reagent
4. Decane	ACS grade decane	Same as above	Same as above

(Continued)

Table 1.1 (Continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Calibration gases for analysis			
1. Oxidation catalyst efficiency check calibration gas	Gas mixture with nominal concentration of 1 percent methane	Visually check specifications	Return to supplier, order from new supplier
2. FID linearity and NMO calibration gases	Three gas mixture standards with nominal propane concentrations of 20, 200 and 3000 ppm	Check specifications, compare against NBS-SRM propane or previous calibration gas	Return to manufacturer
3. CO_2 calibration gases	Three gas mixture standards with nominal CO_2 concentrations of 50, 500, and 1 percent in air	Check specifications, compare against previous calibration gases or NBS-SRM No. 2622 (2% CO_2 in N_2) diluted with 20% zero oxygen	Return to manufacturer
NMO analyzer system check calibration gas			
1. Propane mixture	Gas mixture containing (nominal) 50 ppm CO, 50 ppm CH_4, 2% CO_2, and 20 ppm C_3H_8 in air	Check specifications, conduct calibration check which should be within 5% of initial NMO response factor	Return to manufacturer, check new gas
2. Hexane gas standard	Gas mixture containing (nominal) 50 ppm hexane in air	As shown above	As shown above
3. Toluene gas standard	Gas mixture containing (nominal) 20 ppm toluene	As shown above	As shown above
4. Methanol gas standard	Gas mixture containing (nominal) 100 ppm mehanol in air	As shown above	As shown above

2.0 CALIBRATION OF APPARATUS

Calibration of the apparatus is one of the most important functions in maintaining data quality. The detailed calibration procedures included in this section were designed for the sampling equipment specified in Method 25 and described in the previous section. The calibration of the analytical equipment is described in the section detailing the analytical procedures, Section 3.17.5. Table 2.1 at the end of this section summarizes the quality assurance functions for the calibrations addressed in this section. All calibrations including the analytical equipment should be recorded on standardized forms and retained in a calibration log book.

2.1 Sample Metering and Volume Systems

2.1.1 *Sample Tank Volume* - The volume of the gas sampling tanks used to for sampling and as intermediate collection vessels must be determined as follows:

1. Mark or number each tank so that it is uniquely identified.
2. Weigh each tank empty to the nearest 5 g.
3. Fill the tank with distilled or deionized water and reweigh to nearest 5 g.
4. Record the data on Figure 2.1 or similar form and calculate the sample tank volume.
5. Alternatively, the volume of the water used to fill each tank may be determined to the nearest 5 ml. It is extremely important that all the water is removed from the tank after calibration.

2.1.2 *Volume of Sampling Train from Probe Tip to Sample Tank Valve* - The volume of the sampling train from the probe tip to the sample tank valve must be determined as follows:

1. Assemble the sampling train in the same manner as it will be used in the field.
2. Leak check the sampling system as described below in Section 2.1.3.
3. After the train passes the leak check, attach a sample tank that has been calibrated and is full of air at ambient pressure to the sampling system.
4. With the inlet still plugged from the leak check, open the sample tank valve, flow control valve, sample valve, and purge valve to ensure that the tank is at ambient pressure. Note that the mercury U-tube manometer is reading zero.
5. Shut the sample tank valve and start the purge pump (or other pump of the tester's choice).
6. After approximately 2 min, shut the purge valve and then turn off the purge pump.
7. Open the sample tank valve and read and record the vacuum.
8. Record these data and the barometric pressure on Figure 2.2 or similar form and then calculate the volume of the sampling train.
9. Repeat steps 4 through 8 twice. The calculated sampling train volume used for the leak checks during testing will be the average of the three calculated volumes. If the equipment is similar or the components are interchangeable for all the Method 25 sampling systems used, the volume from the probe tip to the sample tank valve only needs to be determined for one system.

Sample Tank No.	Date Calibrated	Initial Weight/Volume, g/ml*	Final Weight/Volume, g/ml*	Sample Tank Volume, ml*	Calibrated By, initials

*Weight measured to the nearest 5 g or volume measured to the nearest 5 ml.

Figure 2.1. Sample tank and intermediate collection vessel volume determination.

Calibrated By ________________ Date Calibrated ________________

System Assembled ________________ System Leak Checked ________________

Tank No. ________________ Tank Volume, ml ________________

Barometric Press., mm Hg _________

Are sampling train components similar and/or interchangeable? ____________

Will the calculated sampling train volume be used for all trains? _________

Run No.	Vacuum Reading, mm Hg	Calculated Volume, ml	Remarks
1			
2			
3			

Average calculated volume of sampling system __________ ml

$$\text{Calculated volume, ml} = \frac{\text{Vacuum reading, mm Hg} \times \text{Tank volume, ml}}{\text{Barometric press, mm Hg} - \text{Vacuum reading, mm Hg}}$$

$$\text{Calculated volume, ml} = \frac{(\qquad\qquad) \times (\qquad\qquad)}{(\qquad\qquad) - (\qquad\qquad)} = \underline{\qquad\qquad} \text{ ml}$$

$$\text{Average volume, ml} = \frac{\text{Run 1} + \text{Run 2} + \text{Run 3}}{3} = \underline{\qquad\qquad} \text{ ml}$$

Figure 2.2. Determination of sample train volume from probe tip to sample valve.

2.1.3 *Rotameters* - Two rotameters are needed, one for purging the sampling system and a second for controlling the rate of sample collection. Since the sampling rotameter is used to determine flow rate and maintain a constant sampling rate, it must have an accuracy of ± 10% for the flow rate used (60 to 100 cc/min) and a precision of ± 5% over its range. The rotameter used to purge the sampling train serves only as an indicator of the flow rate and its readings are not used in any emission calculations; therefore, the accuracy of ± 10% and precision of ± 5% do not apply after its initial calibration.

Initial Calibration - Both rotameters should be calibrated as part of the sampling system when first purchased and at any time the posttest check yields a calculated sample volume that is not within 10% of the actual sample volume (for sampling rotameter only) or erratic behavior is noted (for both rotameters). A calibrated wet test meter, calibrated dry gas meter, or a properly sized bubble meter should be used to calibrate the rotameters.

Before its initial use in the field, each rotameter should be calibrated as part of the entire sampling system as described below.

1. Leak check the rotameter as part of the sampling system as follows:
   a. Temporarily attach a suitable rotameter (e.g., 0-40 cm^3/min) to the outlet of the purge system.
   b. Plug the inlet to the probe. Shut the sample tank valve and open the flow control valve, purge valve, and the sample valve. Evacuate the entire sampling system to 10 mm Hg.
   c. Note the flow rate as indicated by the rotameter.
   d. A leak of $\leq$0.02 L/min must be obtained; leaks >0.02 L/min must be eliminated.
   e. Close the purge valve and turn off the pump.
   f. Note the vacuum reading.
   g. Wait five minutes and take another vacuum reading.
   h. If the pressure has changed by more than 20 mm Hg, the leak should be found and corrected.
2. Attach a wet test meter, bubble meter, or calibrated dry gas meter to the inlet of the probe.
3. Run the pump for 15 minutes with the flow rate set in the midrange (80 cc/min) to allow the pump to warm up and to permit the interior surface of the wet test meter to become wet.
4. Collect the information required in the forms provided [Figure 2.3A (English units) or Figure 2.3B (metric units)] using sample volumes equivalent to at least five revolutions of the test meter or 10 minutes, whichever is greater. Three independent runs must be made covering the top, middle, and bottom of the flow rate range (i.e., 60, 80, and 100 cc/min).
5. Calculate the Y_i for each run as shown on the data forms. The Y should be in the range of 0.9 to 1.1 and the values of Y_i should be in the interval Y ± 0.05Y, where Y is the average for three runs. If not, recalibrate, repair, or replace the rotameter. Otherwise, the Y (calibration factor) is acceptable and is to be used for future checks and subsequent test runs. Alternatively, if the Y_i's are acceptable and the Y is outside the range of 0.9 to 1.1, the rotameter may be remarked to reflect the corrected readings. The corrected readings will then be used for testing and future calibrations. The completed form should be forwarded to the supervisor for approval, and then filed in the calibration log book.

Date ________ Calibrated by ________ Meter system no. ________ Primary meter no. ________
Barometric pressure, P_m = ________ in. Hg Ambient temperature ________ °F
Type of primary meter: wet test ________, dry gas ________, or bubble meter ________

Primary meter readings					Rotameter readings							
Initial reading (V_{pi}),[a] ft^3	Final reading (V_{pf}),[a] ft^3	Initial temp, °F (t_{pi}) °F	Final temp, °F (t_{pf}) °F	Press drop (D_p)[c] in. H_2O	Initial reading (V_{si}),[b] ft^3 or ft^3/min	Final reading (V_{sf})[b] ft^3 or ft^3/min	Initial temp (t_{si}) °F	Final temp (t_{sf}) °F	Press drop (D_s),[c] in. H_2O	Time min (θ),[d] min	Calibration factors	
											(Y_i),[e]	(Y)

[a] Volume passing through the meter using the initial and final readings; requires a minimum of at least five revolutions of the meter.

[b] Volume passing through the meter using the initial and final readings or the indicated flow rate using the initial and final flow rate setting.

[c] Pressure drop through the meter used to calculate the meter pressure.

[d] The time it takes to complete the calibration run.

[e] With Y defined as the average ratio of volumes for the primary meter compared to the flowmeter calibrated, Y must be 0.9 to 1.1 and $Y_i = Y \pm 0.05Y$ thus:

$$Y_i = \frac{(V_{pf} - V_{pi})[(t_{si} + t_{sf})/2 + 460°F][P_m + (D_p/13.6)]}{[(V_{sf} + V_{si})/2]\theta[(t_{pi} + t_{pf})/2 + 460°F][P_m + (D_s/13.6)]} \text{(Eq. 2-3)}, \quad Y = \frac{Y_1 + Y_2 + Y_3}{3} = \underline{\qquad} \text{(Eq.2-4)}$$

Figure 2.3A. Rotameter calibration data form (English units).

Date __________ Calibrated by __________ Meter system no. ________ Primary meter no. __________

Barometric pressure, P_m = ____________ mm Hg Ambient temperature __________ °C

Type of primary meter: wet test ________, dry gas ____________, or bubble meter ______________

Primary meter readings					Rotameter readings							
Initial reading (V_{pi}),[a] m^3	Final reading (V_{pf}),[a] m^3	Initial temp, °F (t_{pi}) °C	Final temp, °F (t_{pf}) °C	Pres drop (D_p)[c] mm H_2O	Initial reading (V_{si}),[b] m^3 or m^3/min	Final reading (V_{sf})[b] m^3 or m^3/min	Initial temp (t_{si}) °C	Final temp (t_{sf}) °C	Press drop (D_s),[c] mm H_2O	Time min (θ),[d] min	Calibration factors (Y_i),[e]	(Y)

[a] Volume passing through the meter using the initial and final readings and requires a minimum of at least five revolutions of the meter.

[b] Volume passing through the meter using the initial and final readings or the indicated flow rate using the initial and final flow rate setting.

[c] Pressure drop through the meter used to calculate the meter pressure.

[d] The time it takes to complete the calibration run.

[e] With Y defined as the average ratio of volumes for the primary meter compared to the rotameter calibrated, Y must be 0.9 to 1.1 and $Y_i = Y \pm 0.05Y$ thus,

$$Y_i = \frac{(V_{pf} - V_{pi})[(t_{si} + t_{sf})/2 + 273°K][P_m + (D_p/13.6)]}{[(V_{sf} + V_{si})/2]\theta[(t_{pi} + t_{pf})/2 + 273°K][P_m + (D_s/13.6)]} \text{ (Eq. 2-7)}, \quad Y = \frac{Y_1 + Y_2 + Y_3}{3} = \underline{\quad\quad} \text{ (Eq. 2-8)}$$

Figure 2.3B. Rotameter calibration data form (metric units).

Posttest Calibration Check - After each field test series, calculate the volume of sample that should have been collected during the sample run and compare the results with the actual volume collected. If the calculated volume and the actual volume collected are within 10% of each other on the average for the three runs, no recalibration is needed. If the average sample volumes are not within 10%, the sampling rotameter should be recalibrated. When either the sampling rotameter or purge rotameter exhibits erratic behavior during sampling or purging of the system, it should be recalibrated. Performance of a posttest calibration does not necessitate changes in the emission calculations.

2.3 Thermocouples

The thermocouples on the sample probes and the filter heating system should be initially compared with a mercury-in-glass thermometer that meets ASTM E-1 No. 63C or 63F specifications:

1. Place the thermocouples to be calibrated and the mercury-in-glass thermometer in a bath of boiling water. Compare the readings after the bath temperature stabilizes and then record them on the calibration data form, Figure 2.4 or equivalent.
2. Allow both the thermocouple and reference thermometer to come to room temperature. Compare the readings after the temperature readouts stabilize.
3. The thermocouple is acceptable if the values agree within 3°C (5.4°F) at both points.
4. Prior to each field trip, compare the temperature reading of the mercury-in-glass thermometer at room temperature with that of the thermocouple that is part of the metering system. If the values are not within 3°C (5.4°F) of each other, replace or recalibrate the thermocouple.
5. No posttest calibration of the thermocouples is required unless they demonstrated erratic behavior during the sampling.

2.4 Barometer

The field barometer should be adjusted initially and before each test series to agree within 2.54 mm (-0.1 in.) Hg with a mercury-in-glass barometer or with the pressure value reported from a nearby National Weather Service Station and corrected for elevation. The tester should be aware that the National Weather Service readings are normally corrected to sea level; uncorrected readings should be obtained. The correction for the elevation difference between the weather station and the sampling point should be applied at a rate of -2.5 mm Hg/30 m (-0.1 in. Hg/100 ft) elevation increase, or vice versa for elevation decrease.

2.5 Absolute Pressure Gauge

The absolute pressure gauge should be calibrated against a mercury U-tube manometer upon receipt and every quarter thereafter or upon erratic behavior. Attach the the absolute pressure gauge and mercury U-tube manometer to a "T" connection with a vacuum pump. Compare the readings at atmospheric pressure. Pull a vacuum of 10 mm Hg of absolute pressure. Pressure readings should agree within 3 mm Hg. If this criteria is not met, make adjustments and repeat the calibration.

Date	Reference thermometer type	Calibrated thermocouple			Measured values						Calibrator's initials
					Ambient temperature			Boiling water			
		type	use	no.	refer[a]	calibr[b]	differ[c]	refer[a]	calibr[b]	differ[c]	

[a] Temperature reading of the reference thermometer in °C or °F.

[b] Temperature reading of the thermocouple being calibrated in °C or °F.

[c] Difference between the reference thermometer and the calibrated thermocouple. This difference must be less than 3°C (5.4°F) for than initial calibration and 6°C (10.4°F) for the calibration check.

Figure 2.4. Thermocouple calibration form.

Table 2.1. ACTIVITY MATRIX FOR CALIBRATION OF EQUIPMENT

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Sample tank volume	Within 5 g or 5 ml	Calibrate initially and when not within 10% of calculated volume or shows erratic behavior	Repair or replace and recalibrate
Sampling train volume	No limits	Calibrate initially as described in Section 2.1.2	Repeat
Rotameters	$Y = 0.9$ to 1.1 and all $Y_i = Y \pm 0.5\ Y$	Calibrated initially and when calculated volume not within 10% or erratic behavior	Repair, replace, and recalibrate
Thermometers	Within 3°C (5.4°F) of true value	Calibrate initially as a separate component with mercury-in-glass thermometer; check before each test against mercury-in-glass thermometer	Adjust or replace
Barometer	Within 2.5 mm (0.1 in.) Hg of mercury-in-glass barometer or weather station value	Calibrate initially using mercury-in-glass barometer; check before and after each test	Adjust to agree with certified barometer
Absolute Pressure Gauge	Within 3 mm Hg of mercury U-tube manometer	Calibrate initially using mercury U-tube manometer; repeat every quarter or upon erratic behavior	Adjust to agree with mercury U-tube manometer

3.0 PRESAMPLING OPERATIONS

This section addresses the preparation and packing of supplies and equipment needed for the sampling. The pretest sampling checks (Figure 3.1) is a listing of equipment pretest operational checks which should be completed before leaving for the field. The pretest preparations form (Figure 3.2) can be used as an equipment packing checklist. The quality assurance activities for the presampling operations are summarized in Table 3.1 at the end of this section.

3.1 Preliminary Survey

The preliminary survey may be needed to properly design the final emission sampling and analytical protocol. Preliminary measurements may include determining the stack dimensions, the flue gas moisture, stack pressure, and stack temperature, if the concentration of organics is to be determined on the basis of stack conditions. Other measurements which may be made, depending upon the requirements of the applicable regulation and the source operations, include a flow rate determination, velocity check, and stack gas temperature range. The tester must determine these requirements and use the proper test methods to establish these data (i.e., EPA Methods 1 through 4).

One of the primary concerns for any organic sampling program must be safety. The tester should always question the facility representative concerning general plant safety requirements and safety in regard to sampling at the selected sampling site. Every sampling and analysis protocol should address the safety considerations involved in performing the protocol. Because there are numerous safety considerations involved in organic sampling, it is beyond the scope of this Handbook to discuss each one in detail. However, it cannot be over-emphasized that the tester must always be aware of the safety hazards.

Figure 3.3 may be used to collect preliminary survey data.

3.2 Checking and Calibrating the Apparatus

3.2.1 *Sampling System* - The Method 25 sampling train (see Figure 1.1) is commercially available or can be manufactured in-house if the apparatus complies with specifications in the EPA Test Method (see Section 3.17.10). These specifications and other performance criteria are discussed in greater detail in Section 3.17.1.

Heated Probe - The probe's thermocouple should have been initially calibrated as described in Subsection 3.17.2.3. Prior to each field test, the thermocouple should be attached to the temperature readout device and the probe thermocouple reading should be compared with a reference thermometer reading at the ambient temperature. If the values are not within 3°C (5.4°F) of each other, replace or recalibrate the thermocouple.

The probe should be cleaned with acetone or methylene chloride, nitric acid, and distilled or deionized water. To dry the probe, turn on the probe heater and purge the probe with UPC air or nitrogen. The objective is to leave the probe free of contamination.

Leak check the probe and check the probe heater system as part of the complete train assembly as described below.

Date ______________________________ Calibrated by ____________________________

Heated Probe

Heater system capable of maintaining heat at probe exit at 129°C? ______ yes ___ no
Thermocouple calibrated against reference thermometer? ____ yes ___ no (within 3°C)
Probe cleaned with soap and water and acetone, then dried? _____ yes ____ no
Probe leak checked with sampling system? _____ yes _____ no

Filter Holder/Heating System

Heater system capable of maintaining heat at filter exit at 121°C? ____ yes ___ no
Thermocouple calibrated against reference thermometer? ____ yes ___ no (within 3°C)
Assembly cleaned with soap and water and acetone, then dried? _____ yes ____ no
Filter assembly leak checked with sampling system? _____ yes _____ no

Condensate Trap

Traps burned at 300°C until acceptable blank level achieved? ____ yes ____ no
Nitrogen placed in trap and trap sealed? _____ yes ____ no

Purge System

Rotameter initially calibrated? _____ yes ______ no (accuracy 10%, precision 5%)
Purge system checked with sampling system and indicated flow? _____ yes ____ no

Sampling Rotameter

Initially calibrated against primary standard? ____ yes _____ no (accuracy 10%, precision 5%)
Calculated sample volume within 10% of actual sample for last test? _____ yes ___ no

Sample Tank

Tank volume calibrated gravimetrically or volumetrically? ____ yes ____ no (± 5 ml)
Tank cleaned and filled with nitrogen? ____ yes ____ no

Barometer or Absolute Pressure Gauge

Calibrated against mercury-in-glass barometer? _____ yes _____ no (± 2.54 mm Hg)

Ambient Thermometer

Initially calibrated against reference thermometer? _____ yes ____ no (± 1°C)

Figure 3.1. Pretest sampling checks.

Apparatus check	Acceptable		Quantity Required	Ready		Loaded and Packed	
	Yes	No		Yes	No	Yes	No
Sampling System *Heated Probe* ______ *Heating checked* *Thermocouple calibrated** *Cleaned** *Leak checked*							
Filter Holder/Heater *Heater checked* *Thermocouple calibrated** *Cleaned** *Leak checked*							
Condensate Trap *Burned & blank checked** *Sealed**							
Purge System *Pulls flow*							
Sampling Rotameter *Calibrated or checked**							
Sample Tanks *Calibrated** *Cleaned**							
Pressure Gauge *Calibrated**							
Ambient Thermometer *Calibrated or checked*							
Sampling Supplies *Dry ice* *Filters* *Free of organic binder**							

*Most significant items/parameters to be checked.

Figure 3.2. Pretest preparations.

I. Name of company ____________ Date ____________
Address ____________
____
Contacts ____________ Phone ____________
____________ ____________
Process to be sampled ____________
____
____
Duct or vent to be sampled ____________
____

II. Process description ____________
____
____
____
____
Raw material ____________
____
Products ____________
____
Operating cycle
Check: Batch ________ Continuous ________ Cyclic ________
Timing of batch or cycle ____________
Best time to test ____________

III. Sampling site
A. Description
Site description ____________
Duct shape and size ____________
Material ____________
Wall thickness ____________ inches
Upstream distance ________ inches ________ diameter
Downstream distance ________ inches ________ diameter
Size of port ____________
Size of access area ____________
Hazards ____________ Ambient temp ________ °F
B. Properties of gas stream
Temperature ________ °C ________ °F, Data source ________
Velocity ____________, Data source ________
Static pressure ________ inches H_2O, Data source ________
Moisture content ________ %, Data source ________
Particulate content ________, Data source ________
Gaseous components

N_2 ________ %	Hydrocarbons (ppm)	Toxics/Acids (ppm)
O_2 ________ %	________	H_2S ________
CO ________ %	________	HCl ________
CO_2 ________ %	________	HF ________
SO_2 ________ %	________	Other ________

Figure 3.3. Preliminary survey data sheet.

(Continued)

Figure 3.3 (Continued)

Hydrocarbon components

__________________ ____________ ppm
__________________ ____________ ppm
__________________ ____________ ppm
__________________ ____________ ppm
__________________ ____________ ppm
__________________ ____________ ppm

C. Sampling considerations
Location to set up sample recovery area ________________
____
Special hazards to be considered ________________________
____
Power available at duct ____________________________
Power available for GC ____________________________
Plant safety requirements ___________________________
____
Vehicle traffic rules ______________________________
____
Plant entry requirements ___________________________
____
Security agreements ______________________________
____
Potential problems _______________________________
____
Safety equipment (glasses, hard hats, shoes, etc.) _____
____

D. Site diagrams. (Attach additional sheets if required).

IV. On-site collection of preliminary survey samples

A. Evacuated tank
Tank have been cleaned, heated in furnace and purged with nitrogen? ______________________________
Tank evacuated to the capacity of pump? ______________
Filter end of probe placed at center of stack, probe purged and sampled collected into flask until flask is at stack pressure? ______________________________
Stopcocks closed and taped? ______________________
Duct temperature and pressure recorded? ______________

B. Purged flasks
Flasks cleaned and purged with nitrogen? ______________
Filter end of probe placed into stack, sample purged for 2 to 5 min and then stopcocks closed? ______________
Stopcocks taped to prevent leakage? ________________
Duct temperature and pressure recorded? ______________
Stability and adsorption checks conducted? ____________

(Continued)

Figure 3.3 (Continued)

C. Quality assurance performance audit samples
Range of emissions to order proper range of performance audit samples __________ inlet ppm _________ outlet ppm
Address to send audit samples ________________________

D. Bulk samples and screening techniques
Bulk emission sample(s) collected?____________________
Bulk liquid sample(s) collected?______________________
Detector tubes or other screening techniques used?_______

E. Safety with respect to sample collection
Can gases be exhausted through purge system? ___ yes ___no
Can electrical service be used? _____ yes _____ no
Can heated probe and heated filter be used? ____ yes ___no
Can thermocouple be used? ______ yes ______ no

F. Emission results must be reported in terms of:
ppmC at standard conditions ____________________
ppmC at stack conditions ______________________
ppmC at standard conditions corrected for CO_2 ________
ppmv as a related solvent at standard conditions ______
ppmv as a related solvent at stack conditions ________
pounds per hour of carbon ______________________
pounds per hour of related solvent _______________

Filter Holder and Heating System - The filter holder assembly's thermocouple should have been initially calibrated as described in Subsection 3.17.2.3. Prior to each field test, the thermocouple should be attached to the readout and the probe thermocouple reading should be compared with a reference thermometer reading at the ambient temperature. If the values are not within 3°C (5.4°F) of each other, replace or recalibrate the thermocouple.

The filter holder/heating assembly should be cleaned with acetone or methylene chloride, nitric acid, and distilled or deionized water. To dry the filter holder, turn on the probe heater and purge the filter holder with UPC air or nitrogen. The objective is to leave the assembly free of contamination.

Leak check the filter holder assembly and check the filter heater system as part of the complete train assembly as described below.

Condensate Trap - Before its initial use and after each subsequent use, a condensate trap should be thoroughly cleaned and checked to insure that it is not contaminated. Both cleaning and checking can be accomplished by installing the trap in the condensate recovery system and heating to 300°C while performing a system background test (described in Section 3.17.5.2.1). A trap may be considered clean when its effluent concentration is below 10 ppm. Clean or "blanked" traps should be filled with nitrogen and sealed to prevent contamination and corrosion. If a trap cannot be properly "blanked," it should be discarded. The history of the trap should also be tracked. It is recommended that traps previously used for inlet sampling be used only for inlet sampling thereafter.

Purge System - The purge system must be capable of purging the probe and filter holder assembly at a rate of 60 to 100 cc/min. Upon initial receipt, the rotameter should be calibrated as described in Section 3.17.2. After the initial calibration, the rotameter and pump should be checked prior to each field test to ensure that it is capable of purging the sampling system at the rate indicated above. The rotameter for the purge pump is used only as a flow indicator during the field test.

To check the operations of the sampling system, the probe, filter holder/heating assembly, and purge system should be assembled in the same manner that it will be used in the field with the exception that (1) no filter is needed and (2) the tank will not be attached. Check the system as follows:

1. Turn on the probe and the filter holder heaters.
2. With the inlet to the probe open, turn the sample tank valve off, turn the purge pump on, set the flow rate to about 80 cc/min, and allow air to be drawn through the system until the operating temperatures are met.
3. After the probe and filter holder comes to the proper temperatures (probe 129°C and filter 121°C), plug the inlet to the probe and conduct a leak check as described in Subsection 1.1.10. If the system does not pass the leak check, repair or replace the faulty component(s) and repeat the leak check until it is acceptable.

Sampling Rotameter - The sampling rotameter must be calibrated (1) initially as described in Subsection 2.1.3, (2) when the posttest calculated volume from the previous field test is not within 10% of the actual sample volume collected and (3) any time the rotameter exhibits erratic behavior. If the rotameter is acceptable using the criteria above, the tester may attach the purge pump after the sample tank valve and ensure that the rotameter does not exhibit erratic behavior by pulling a flow of about 80 cc/min. If erratic behavior is exhibited, the rotameter should be cleaned and recalibrated.

Sample Tanks - Each sample tank must be initially calibrated as described in Section 3.17.2. After the initial calibration, it should be visually checked prior to each field test to ensure that there are no dents that would effect the total sample volume. Each tank must also be flushed with UPC air until there is no response on the NMO analyzer. After a tank is free of hydrocarbon contamination, it should be evacuated and filled with nitrogen to a pressure of approximately 10 mm Hg above atmosphere. The nitrogen will prevent contamination and corrosion.

Mercury Manometer or Absolute Pressure Gauge - If a mercury manometer is used, then a barometer will be required. No calibration is required for a mercury manometer. The manometer or absolute pressure gauge should be leak checked as part of the assembly as described above. The barometer and/or absolute pressure gauge should be calibrated against a mercury-in-glass barometer as described in Subsection 3.17.2.4. If it does not agree within 2.54 mm Hg, it should be corrected or replaced.

Thermometer for Ambient Temperature - A thermometer is needed to measure the ambient temperature. If the thermometer is a mercury-in-glass thermometer with a sensitivity of 1°F, the thermometer should be initially check against a mercury-in-glass ASTM thermometer as described in Section 3.17.2. After the initial calibration, the thermometer should only be visually checked to ensure that it is not broken. If a thermocouple is used to measure the ambient temperature, it should be checked at ambient temperature against a reference thermometer and should agree with 1°C.

3.2.2 *Sampling Supplies* - The following supplies are needed for sampling:

Crushed Dry Ice - Crushed dry ice is needed to cool the condensate trap during sampling for better collection of organics and to keep it cool until analysis. There is no specification on the dry ice.

Filters - Glass fiber filters, without organic binder are needed to remove organic particulate matter from the gas stream during sample collection. Typically, the filters used for EPA Method 5 sampling are satisfactory, if no organic binders are present. If organic binders are present, they may be released during testing and positively bias the results. If the tester is not certain about the presence of organic binders in a glass fiber filter, it should be placed in a furnace at 300°C for 2 hours to remove any organic binders present. This procedure, however, may make the filter more brittle resulting in a greater need for caution in handling. A check on the amount of organic binder lost during heating can be determined by following the procedures described in Subsection 1.3.1.

3.3 Packing the Equipment for Shipment

The sampling system is relatively small, made predominately of stainless steel, and therefore rugged with the exception of the rotameters, pumps, and pressure gauges. The filter holder system and probe may be packed separately or as a unit. The other components should be packed conveniently and securely in labeled containers (as to contents) for ease of identification in the field. Polyethylene foam can be used to cushion the components. Also, the tanks should be secured so they do not become dented.

Table 3.1. ACTIVITY MATRIX FOR PRESAMPLING PREPARATION

Operation	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Apparatus Check and Calibration			
Heated probe	Leak free, cleaned, capable of heating to 129°C with calibrated thermocouple	Clean with soap and water, then acetone; calibrate thermocouple against reference thermometer; conduct heater check and leak check with assembly	Repeat cleaning, calibration, and/or heater and leak checks
Filter holder	Leak free, cleaned, capable of heating to 121°C with calibrated thermocouple	As above	As above
Condensate trap	Acceptable blank level	Check as described in Subsection 3.17.5.__	Repeat burnout and repeat blank check or replace and repeat
Purge system	Capable of purging at rate of 80 cc/min	Check as part of sampling assembly	Repair and repeat
Rotameter	Calibrated	Calibrate as shown in Section 3.17.2	Clean and recalibrate
Sample tank	Volume calibrated to ± 5 ml and clean	Calibrate and clean as described in Subsection 3.17.1.1	Recalibrate and/or reclean
Pressure gauge	Range 0 to 900 mm Hg within 2.54 mm Hg	Check against mercury-in-glass barometer	Adjust and repeat calibration
Sampling Supplies			
Dry ice	No specifications	Not applicable	Not applicable
Filters	Glass fiber with no organic binders	Check or heat filter	Replace
Packing Equipment for Shipment	Packed in secure container	Before field trip, pack in shipping container	Repack

4.0 ON-SITE MEASUREMENTS

On-site activities include transporting the equipment to the test site, unpacking and assembling the equipment, sampling for total gaseous nonmethane organics, and recording the data. The associated quality assurance activities are summarized in Table 4.1 at the end of this section.

4.1 Transport of Equipment to the Sampling Site

The most efficient means of transporting the equipment from ground level to the sampling site (often above ground level) should be decided during the preliminary site visit or by prior correspondence. Care should be taken to prevent damage to the equipment or injury to test personnel during the moving. A clean "laboratory" type area free of excessive dust and organic compounds should be located and designated for preparing the tanks, traps, and filter holder, and for sample recovery.

4.2 Preliminary Measurements and Setup

It is recommended that a preliminary survey be conducted prior to sampling and analysis, unless adequate prior knowledge of the source and/or information is available. Testing must be conducted at the proper sampling locations and during the proper process and control equipment operating cycles or periods. The tester should refer to Subsection 3.17.3.1 regarding the information that is typically needed to establish the proper sampling and analysis protocol. The accuracy of sampling and analysis following handling and transportation of the sampling system to and from the sampling site is determined using a cylinder gas audit.

One of the primary concerns for any organic sampling program must be safety. The tester should always question the facility representative concerning general plant safety requirements and safety in regard to sampling at the selected sampling site. Every sampling and analysis protocol should address the safety considerations involved in performing the protocol. Because there are numerous safety considerations involved in organic sampling, it is beyond the scope of this Handbook to discuss each one in detail. However, it cannot be over-emphasized that the tester must always be aware of the safety hazards.

4.3 Sampling

Because of the unlimited variations in sampling organic compounds from potential source types, only the more general common situations and problems are addressed in this section. Both required and recommended quality assurance/control checks and procedures are provided to assist in the collection of acceptable quality data and to assess the accuracy of the sampling and analysis.

On-site sampling includes the following steps:

1. Conducting preliminary measurements and setup of the recovery area.
2. Preparation and setup of the sampling system.
3. Connection of electrical service and leak check of the sampling system.
4. Heating the probe and filter to proper temperature.
5. Insertion of the probe into the duct and sealing the duct.
6. Purging of the sampling system.
7. Constant rate sampling.
8. Recording data.

9. Recovering the sample and its transportation to the laboratory.

4.3.1 *Preliminary Measurements and Setup of Recovery Area* - All test personnel should be knowledgeable of the plant safety requirements. This includes areas that should not be entered and whether the plant will allow the use of electrical service in the sample collection area. The risk to test personnel of being in process areas handling organic compounds, removing gases from these processes, and venting sample gases into the sample collection area may be significant. The plant may require that no electrical service be used and/or that the sample gases removed from the process during the pretest purging of the sampling system be adsorbed onto some vehicle such as a resin or charcoal or vented back into the process.

After all testing personnel are familiar with plant safety requirements and all potential safety hazards, preliminary measurements and setup may begin. The sampling site should be checked to ensure that adequate electrical service is available (if allowed). The stack dimensions are measured and recorded (if applicable) on a field sampling data sheet such as the one shown if Figure 4.1. If the concentration of the organics are to be determined on any basis other than ppmC at standard conditions, the corresponding preliminary measurements should be made at this time. Moisture content of the stack cannot be measured by Method 25. Therefore, if the final emissions are to be presented in terms of stack concentration or mass emission rate, or are to be corrected to a CO_2 concentration, or to an equivalent solvent basis, Methods 1 through 4 will most likely need to be conducted simultaneously with Method 25. Prior to final sampling, the tester must determine on what basis the final results are to presented.

If the emissions are to be presented in terms of a mass emission rate, the flow rate of the stack gas, including its moisture content, must be determined. In this case, it is preferable that the sampling location be selected in accordance with Method 1 (or Method 1A, if applicable). If this is impractical, it should be selected to minimize flow disturbances. The number and locations of sampling points for the velocity traverse are selected according to Method 1 (see Section 3.0.1 of this Handbook); the traverse is conducted according to Method 2 (see Section 3.1 of this Handbook). Note: The Method 25 sampling is conducted at a single point of average velocity. If it is unsafe to conduct a preliminary velocity traverse or a traverse is not required, the sampling may be conducted at the center of the duct or at a point at least 3 feet into the duct (whichever is less). The port must be sealed well and there must be no reason to suspect that the emission concentration is not uniform across the stack.

Method 25 requires constant rate sampling; the sampling rate is not changed with regard to the flue gas flow rate. However, if the emissions are presented on a mass emission basis, the flue gas flow rate must be measured during each Method 25 sample run and the corresponding flow rate used to determine the mass emission rate for that run.

Select a total sampling time greater than or equal to the minimum sampling time specified in the applicable subpart of the regulation or other applicable emission standard. The data will be recorded at 5-minute intervals.

A clean "laboratory" type area should be found to load the filter, recover the samples, conduct orsat analyses, prepare and recover the moisture sampling train, and to store other sampling equipment. This area should be free of excess dust or high levels of organics. Because of the relatively small size of the Method 25 sampling equipment and the nature of Method 25 sample recovery, the stack location can often be used as the recovery area.

Company Name ______________________________ Run No. __________
Address ______________________________ Date __________
Sampling Location ______________________________ Start Time __________
Tank No. ____ Trap No. _____ Sampling Train No. _____ Finish Time __________
Thermocouple No. ______ Barometer No. _______ Operator __________
Vacuum Gauge No. _____
Tank Volume ________ liters Train Volume ____________ cc
Calculated Allowable Change p ___________ cm Hg

TANK PARAMETERS

	Barometric Pressure		Tank Temp.		Tank Pressure mm Hg		Leak Check (cm Hg/min)	
Parameter	in. Hg	mm Hg	°F	°C	Gauge	Absolute	Tank	System
Pretest								
Posttest								
Final Pressure								

SAMPLE DATA

Clock Time	Tank Vacuum or Gauge Pressure, in. Hg (mm Hg)	Flow Meter Setting, cc/min	Sample Gas Temp., °F (°C)	
			Probe Exit	Filter Inlet

Figure 4.1. Field sampling data form.

4.3.2 *Sampling System Preparation* - Sampling system preparation includes (1) leak checking the tanks, (2) assembling the sampling train, and (3) leak checking the entire sampling system. This preparation should be conducted as follows:

1. The sample tanks should be evaluated to 10 mm Hg or less absolute pressure. The pressure must be measured with a mercury U-tube manometer or absolute pressure gauge capable of measurement within 1 mm Hg. Record the tank pressure on the field sampling data form (Figure 4.1 or similar form). Close the sample tank valve and allow the tank to sit for 60 minutes. The tank pressure should then be rechecked using the same pressure gauge and this pressure recorded. If after 60 minutes no noticeable change (less than 4 mm Hg) has occurred in the tank pressure, the tank is acceptable for testing. The tank evacuation and leak check may be conducted either in the laboratory or the field.
2. Just before sampling train assembly, measure the tank vacuum using a mercury U-tube manometer or absolute pressure gauge capable of measurement to within 1 mm Hg. Record this pressure, the ambient temperature, and the current barometric pressure on the field sampling data form.
3. Close the sample tank valve and assemble the sampling system as shown in Figure 1.1.
4. Immerse the condensate trap body in dry ice. The point where the inlet tube joins the trap body should be 2.5 to 5 cm above the top of the dry ice.
5. After assembling the sampling train, plug the probe tip, and make certain that the sample tank valve is closed.
6. Turn on the purge vacuum pump, and evacuate the sampling system from the probe tip to the sample tank valve to an absolute pressure of 10 mm Hg or less and record the pressure on the field sampling data form.
7. Close the purge valve, turn off the pump, wait 5 minutes, and recheck the indicated pressure and record this reading. The change in the absolute pressure of the tank during the 5-min period is the measured pressure change (delta P). Note: A less sensitive pressure gauge, which is standard on commercially available equipment and reads pressure to the nearest 0.5 in. Hg, can be used for indication of the vacuum during testing, but cannot be used for measuring the tank pressure and for the leak check procedure. The tester must conduct the leak check by attaching the mercury U-tube manometer or pressure gauge at the probe inlet.

   The sample tank must remain closed. If opened, the extremely large volume of the tank compared to the sample system makes detecting a small leak extremely difficult.
8. Calculate the maximum allowable pressure change based on a leak rate of 1 percent of the sampling rate using the equation below. Record it on the field sampling data form. This should be compared to the measured P from Step 7 which must be less than or equal to the allowable P calculated in Equation 4-1.

$$P = 0.01 \frac{F P_b \theta}{V_t} \qquad \text{Eq. 4-1}$$

Where:

P = Allowable change, cm Hg,
F = Sampling flow rate, cc/min,
P_b = Barometric pressure, cm Hg,
θ = Leak check period, min, and
V_t = Sampling train volume from probe tip to tank volume, cc.

9. If the measured pressure change exceeds the calculated limit, correct the problem before beginning sampling.
10. Mark the probe such that when placed in the stack, the nozzle will be at a point of average velocity. Alternatively, if the flow cannot be measured or does not have to be measured, mark the probe such that the nozzle will be placed at the center of the duct or a point at least three feet into the duct (whichever is less).
11. Calculate the sampling rate for the test by dividing sampling time (minutes) into 80% of the sample tank volume (cc). The resulting flow rate, in cc/min, will be used for the testing and will provide a margin of error.
12. Complete all remaining entries on the field sampling data form that can be completed prior to the start of the test run. This includes all facility, sampling train component, and run information plus the barometric pressure.

4.3.3 *Constant Rate Sampling* - Sampling must be conducted at a constant rate (± 10%) over the duration of the sampling period at a rate of between 60 and 100 cc/min. Follow the steps below to obtain a constant rate sample:

1. Unplug the probe tip, and place the probe into the stack such that the probe is perpendicular to the duct or stack axis and the probe tip (or nozzle) is located at a point of average velocity with the tip facing away opposite of the direction of gas flow. For ducts with a negative static pressure, sufficiently seal the sampling port to prevent air in-leakage around the probe.
2. Set the probe temperature controller to 129°C (265°F) and the filter temperature controller to 121°C (250°F). Allow the probe and filter to heat for about 30 minutes prior to purging the train.
3. To purge the probe and filter assembly, close the sample valve, open the purge valve, and start the vacuum pump. Set the flow rate between 60 and 100 cc/min, and purge the train with sample gas for at least 10 minutes.
4. When the temperatures at the exit ends of the probe and filter are within their specified ranges, sampling may begin.
5. Check the dry ice level around the condensate trap, and add dry ice, if necessary.
6. To begin sampling, close the purge valve and stop the pump. Record the start time. Open the sample valve and the sample tank valve. Using the flow control valve, set the flow through the sampling train to the calculated flow rate. Record the tank vacuum, flow rate setting, probe temperature, and filter temperature.
7. Adjust the flow rate as necessary to maintain a constant rate (± 10%) throughout the sampling period. Also, adjust the probe and temperature controllers as necessary to maintain the proper temperatures.
8. Record the sample tank vacuum, flowmeter settings, probe temperature, and filter temperature at 5-minute intervals.
9. Sample collection ends when the total sampling time is reached or when the constant flow rate can no longer be maintained due to reduced sample tank vacuum. If sampling must be interrupted before reaching the minimum sampling time (specified in the applicable regulation) because a constant flow rate cannot be maintained, proceed as follows: Record the sample tank pressure and temperature, close the sample tank valve, remove the used sample tank from the sampling train (without disconnecting other parts of the sampling train). Take another evacuated and leak-checked

sample tank, measure and record the new tank vacuum, and attach the new tank to the sampling train. After the new tank is attached to the sampling train, proceed with sampling until the required minimum sampling time has been reached.

10. After sampling is completed, close the flow control valve, and record the final tank vacuum; then record the tank temperature and the barometric pressure. Close the sample tank valve.

The sampling train will be recovered as described below. After the trap and tank are disconnected from the sampling train, both the Sampling System Preparation procedure (steps 1 through 12) and Constant Rate Sampling procedure (steps 1 through 10) are repeated for the next sample run. If the tester feels that the filter will not become plugged during the subsequent sample run, the probe and filter holder/filter assembly including the used filter may be used for any proceeding sample runs.

A cylinder gas performance audit shall be conducted during the sample collection phase of the test. The procedures for collecting the cylinder gas are described in Section 3.17.8. Method 25 requires that each test be audited with two concentrations of cylinder gases. Since the tester must have two sampling trains, a regulator, connecting tubing, and a sampling manifold available for the audit, these items must be included in the test protocol. The collection of the audit gases should be conducted in the same manner as collection of the field samples with the exception that the probe, filter holder, and purge system are not used for collection of the audit gases. If the agency representative is not present at the start of field sampling, the tester should wait until the conclusion of the field test to conduct the audit; it is always preferable to have the agency representative present during the audit. The tester should not break the seal on the audit cylinders until just prior to collecting the audit samples to provide the agency with the maximum opportunity to observe all steps in the collection of the field samples and the audit samples.

4.3.4 *Sample Recovery* - Prior to sample recovery, the flow control valve and the sample tank valve should be closed and the field sampling data form should be completed. If the sampling location is not suitable for conducting sample recovery, the sampling train should be removed to the sample recovery area; otherwise, the sample may be recovered at the sampling location. Samples should be recovered as follows:

1. Disconnect the sample tank from the sampling system. If the less sensitive pressure gauge (see Note in Subsection 4.3.2) was used for sampling, the tank should be immediately attached to the more sensitive gauge (reading to within 1 mm Hg) and the tank pressure recorded.
2. Disconnect the condensate trap at the flowmetering system, and tightly seal both ends of the trap. The probe (from the stack to the filter) is not included as part of the condensate sample.
3. Pack the trap in dry ice during storage and shipping and until the samples are analyzed.
4. Ensure that the condensate trap and the sample tank(s) are properly identified by the test run number and their corresponding identification numbers are properly entered on the field sampling data form. The use of a standardized label is encouraged and is helpful in ensuring consistent identification by the laboratory staff.
5. Label a clean condensate trap and tank as sample blanks. These will be analyzed in the same manner as the field samples and then used to determine the blank level of the sampling system.

4.4 Sampling Problems

Because of the large number and variety of organic processes, it is not possible to discuss all the sampling problems related to Method 25 sampling. Only the seven most common problems will be addressed: (1) high sample gas moisture content and freezing of the trap, (2) no use of electrical service in sampling area, (3) conversion of CO_2 to a carbonate in the trap, (4) use of Method 25 for measuring low levels of organics, (5) sample contamination, (6) sampling and analysis by different companies, and (7) measurement in ducts containing organic droplets.

4.4.1 *High Gas Sample Moisture Content and Freezing of Trap* - If the sample gas has a high moisture content, the small line running from the filter to the condensate trap tends to freeze and the moisture blocks the sample gas flow. For this reason, the trap should clear the dry ice by 2.5 to 5 cm. If freezing of the moisture in the line continues to be a problem, the line may be insulated to improve heat transfer. If the problem persists and sampling cannot be conducted, the tester may place a "preliminary" trap in front of the "primary" trap. The "primary" trap should be placed in an ice bath and the second trap placed in the dry ice bath. After sample recovery, both traps are placed in dry ice until analysis.

4.4.2 *Use Electrical Service Not Permitted for Probe and Filter Heating* - If, for safety reasons, the plant cannot allow the use of electrical service at the sampling site, sampling should be conducted using an in-stack filter. The filter should consist of a stainless steel tube packed with quartz wool, similar to the sampling filter in the original version of Method 25. The condensate trap is connected directly to the in-stack filter, and the sampling system is not purged. Recovery of the condensate trap does not include the in-stack filter. The condensate trap must comply with the revised Method 25 specifications.

4.4.3 *Conversion of CO_2 to Carbonate in Trap* - It has been demonstrated that if ammonia is present in the duct during testing, the CO_2 can be converted to carbonate during testing. This conversion results in a high bias during the analysis of the trap. If this problem occurs, consult the Administrator for an alternative procedure.

4.4.4 *Use of Method 25 for Measuring Low Levels of Organics* - Method 25 was not intended to measure organics at levels below 50 ppmC. However, if the tester has no other options, Method 25 can be used under the following conditions: (1) extreme caution must be used in preparing the traps and tanks and (2) two traps and two tanks should be set aside as field blanks with the analytical results subtracted from the field sample values. This approach will improve measurements at lower level sources, although the precision and accuracy of the method will be poor.

4.4.5 *Sample Contamination* - Sample contamination is a major problem with Method 25 sampling and analysis. Precautions to prevent contamination are listed below:

1. Pretest preparation of the probe, filter holder, traps, and tanks cannot be overemphasized. The probe and filter holder assembly must be cleaned in the manner prescribed to eliminate organic materials. The traps must be burned after analysis to remove any organics. The traps should be filled with nitrogen under pressure and their history should be tracked. It is recommended that no trap previously used for sampling at high

organic levels be used for sampling extremely low levels of organics.

2. All components of the train should be maintained such that the surfaces are never exposed to organics (e.g., oil or other organic compounds), particularly the quick-connects or fittings.
3. All other components, such as the tubing used to connect the audit cylinders to the sampling manifold, must be free of organics.

4.4.6 *Sampling and Analysis by Different Companies* - Because of the small number of laboratories that conduct Method 25 analysis, a large portion of the Method 25 sampling and analysis is conducted by two different companies (i.e., the sampling company and the analytical laboratory). This creates problems in assigning responsibility when audit sample results are not acceptable. If the sampling company wants to check the consistency of the analytical results (especially for low level organic sampling), the tester should obtain extra traps and cylinders from the analytical laboratory. These clean traps and cylinders should not be opened, marked as if they were a sample, and submitted for analysis with the field samples.

4.4.7 *Measurement in Ducts Containing Organic Droplets* - If organic droplets exist in the duct to be sampled, the Method 25 results can be greatly biased. The tester should first try to find another location where the droplets do not exist. If this is not possible, two filters may need to be placed in the system with both being replaced after each sample run. The addition of an in-stack filter should help collect organic droplets and will reduce the loading on the out-of-stack (second) filter.

4.5 Sample Logistics and Packing Equipment

The sampling and sample recovery procedures are followed until the required number of (1) runs are completed, (2) audit samples are collected, and (3) blank samples are labeled. Log all data on the sample recovery and integrity data form, Figure 4.2. At the conclusion of the test:

1. Check all traps and tanks for proper labeling (time, date, location, test run number, and any other pertinent documentation). Be sure that blanks have been set aside and labeled.
2. If possible, make a copy of the field data form(s) in case the originals are lost.
3. Examine all tanks and traps for damage and ensure that the traps are packed in a sufficient amount of dry ice for transport to the base laboratory. Ensure that the containers are labeled properly for shipping to prevent loss of samples or equipment.
4. Review the field sampling data form and any other completed data forms to ensure that all data have been recorded and that all forms are present.

Plant Name ______________________
Sample Recovery Person ______________ Plant Location ______________________

RECOVERED SAMPLES

Run No.	Sample Type Inlet Outlet Audit Blank	Trap No.	Placed on Dry Ice	Tank No.	Date Recovered	Time Recovered	Remarks

LABORATORY CUSTODY

Date of Laboratory Custody ______________
All traps still on dry ice? ______________
Laboratory Person Taking Custody ______________
All samples identifiable? ______________
Remarks ______________________________________

Figure 4.2. Sample recovery and integrity data form.

Table 4.1. ACTIVITY MATRIX FOR ON-SITE MEASUREMENTS

Characteristic	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Preliminary determinations and measurements	If final results on stack concentration basis, determine the moisture content of stack gas	Once each field test; use wet bulb/dry bulb thermometer, Method 4, or sling psychrometer	Complete
	If final results on emission rate basis, determine moisture content and flow rate of stack gas	See above for moisture content; for flow rate, once each field test using Method 1 location, if possible, and Method 2 procedures	Complete
	Determine stack dimensions	Prior to sampling, using tape measure	Complete
	Select sampling time ≥ minimum total sampling time in applicable emission standard; number of minutes between readings should be an integer	Prior to sampling	Complete
Sampling system preparation	Leak check tanks, measure pressure with manometer or absolute pressure gauge to within 1 mm Hg	Prior to sampling	Repair or replace if leaks found
	Assemble sample train as shown in Figure 1.1	Prior to sampling, inspect all connections	Check for leaks, repair system; repeat check
	Mark the probe such that nozzle will be at the point of average velocity; if flow can not or does not need to be be measured, place nozzle in center or or 3 feet into duct	Prior to sampling, determine using stack dimensions	Reposition

(Continued)

Table 4.1 (Continued)

Characteristic	Acceptance limits	Frequency and method of measurement	Action of requirements are not met
Sampling system preparation (continued)	Assemble system as shown in Figure 1.1; no leakage	Before sample collection visually and physically inspect all connections	Check for leaks; repair system; repeat check
	Check dry ice level, and add ice, if necessary	Prior to sampling	Complete
	Close the purge valve and stop the pump; record start time, open sample valve and sample tank, set flow to calculated flow rate and record the tank volume, flow rate setting, probe temp and filter temp	To begin sampling	Complete
	Adjust flow rate to maintain a constant rate and adjust probe and temp controllers as necessary to maintain proper temperatures	During sampling; flow rate should be kept at ± 10% throughout the sampling period	Repeat sampling
	Record sample tank vacuum, flow meter settings, probe temp and filter temp at 5-min intervals throughout sampling	During sampling	Complete
	Calculate sampling rate for the test	Prior to sampling; divide sampling time (min) into 80% of the sample tank volume (cc)	Complete

(Continued)

Table 4.1 (Continued)

Characteristic	Acceptance limits	Frequency and method of measurement	Action of requirements are not met
Constant rate sampling	Adjust probe tip such that probe is perpendicular to stack axis or nozzle is located at a point of average velocity with tip facing opposite of direction of gas flow	Prior to sampling	Reposition; check system for leaks
	Set probe temp to 129°C; set filter temp controller to 121°C allow probe and filter to heat for 30 min	Prior to purging the train; confirm using thermocouples	Adjust heating system
	Purge train, close sample valve, open purge valve, and start vacuum pump	Before sample collection, with sample valve closed	Repeat purge
	To begin sampling, close purge valve and stop pump; open sample valve and sample tank valve	During field test	Repeat sampling
	Sampling must be conducted at a constant rate of ± 10% over duration at a rate between 60 and 100 cc/min	During field test	Repeat sampling
	Sample collection ends when total sampling time is complete or constant flow rate can no longer be maintained due to reduced sample tank volume	During field test	If sampling must be interrupted before reaching the minimum sampling time, see Subsection 4.3.3, Step 9

(Continued)

Table 4.1 (Continued)

Characteristic	Acceptance limits	Frequency and method of measurement	Action of requirements are not met
Constant rate sampling (continued)	After sampling is completed, close the flow control valve; record final tank vacuum and temperature, and barometric pressure; close sample tank valve	Immediately following sampling	Complete
	Conduct cylinder gas performance audit during sample collection phase of test	Collect two audit sample gas concentrations per test; see Section 3.17.8	Complete
Sample recovery	Disconnect sample sample tank from sampling system; record tank pressure within 1 mm Hg	Following sampling	Complete
	Disconnect condensate trap; seal both ends tightly	Following sampling	Complete
	Identify condensate trap and sample tanks by their test run number and sampling location and enter corresponding information on field data form	Following sampling	Complete
	Pack trap samples in dry ice during storage and shipping	Following sample recovery	Complete
	Label clean sample tank and condensate as sample blanks	Following sampling	Complete

(Continued)

Table 4.1 (Continued)

Characteristic	Acceptance limits	Frequency and method of measurement	Action of requirements are not met
Sample logistics	Properly label all bags, containers, etc; record all data on form such as Fig. 4.2	Visually check each sample	Complete the labeling

5.0 POSTSAMPLING OPERATIONS

The postsampling operations include recovery of the condensible organic fraction contained in the condensate trap, analysis of the carbon dioxide collected during condensible organic recovery, and analysis of the noncondensible organic fraction collected during source sampling in the sample tank. These operations are performed for the compliance test samples, the blank sample trap/blank sample tank set, and the audit samples.

Both initial and daily performance checks of the condensible organic recovery system are performed. Initial performance checks include a carrier gas and auxiliary oxygen blank test, an oxidation catalyst efficiency check, and a condensible organic recovery efficiency test. Daily performance checks include a recovery system leak check, a system background test, and an oxidation catalyst efficiency check.

Both initial and daily performance checks and calibration of the NMO analyzer are performed. Initial performance tests include an oxidation catalyst efficiency check, a reduction catalyst efficiency check, an analyzer response linearity check, and a chromatography performance check. The initial calibration is derived from the analyzer linearity check data. Daily calibration of both the CO_2 response and NMO response is performed. Figure 5.18 at the end of this section provides a checklist for monitoring the postsampling operations. Table 5.1 at the end of the section summarizes the quality assurance activities associated with the postsampling operations.

5.1 Initial Performance Tests

Initial performance tests of both the condensible organic recovery system and the NMO analyzer are performed before the systems are first placed into operation, after any shutdown of longer than six months, or after any major modification of the systems.

5.1.1 *NMO Analyzer* (see Figure 5.1)

Oxidation Catalyst Efficiency Test - With both catalysts unheated, perform triplicate analyses of the high level methane standard (nominal 1 percent CH_4 in air). With the oxidation catalyst heated only to its operating temperature, reanalyze the high level methane standard in triplicate. Record data on a data sheet (Figure 5.2) and calculate the oxidation catalyst efficiency using the equation shown the figure. The average response with the oxidation catalyst heated should be less than 1 percent of the average response obtained with both catalysts unheated. If not, replace the oxidation catalyst.

Reduction Catalyst Efficiency Test - With the oxidation catalyst unheated and the reduction catalyst heated to its operating temperature, analyze the high level methane standard in triplicate. Repeat the analysis in triplicate with both catalysts heated to their operating temperatures. Record data on a data sheet (Figure 5.2) and calculate the reduction catalyst efficiency using the equation shown the figure. The responses observed under these two conditions should agree within 5 percent. If not, replace the reduction catalyst.

NMO Response Linearity Test and Initial Calibration - With both catalysts at their operating temperatures, perform triplicate injections of each of the propane

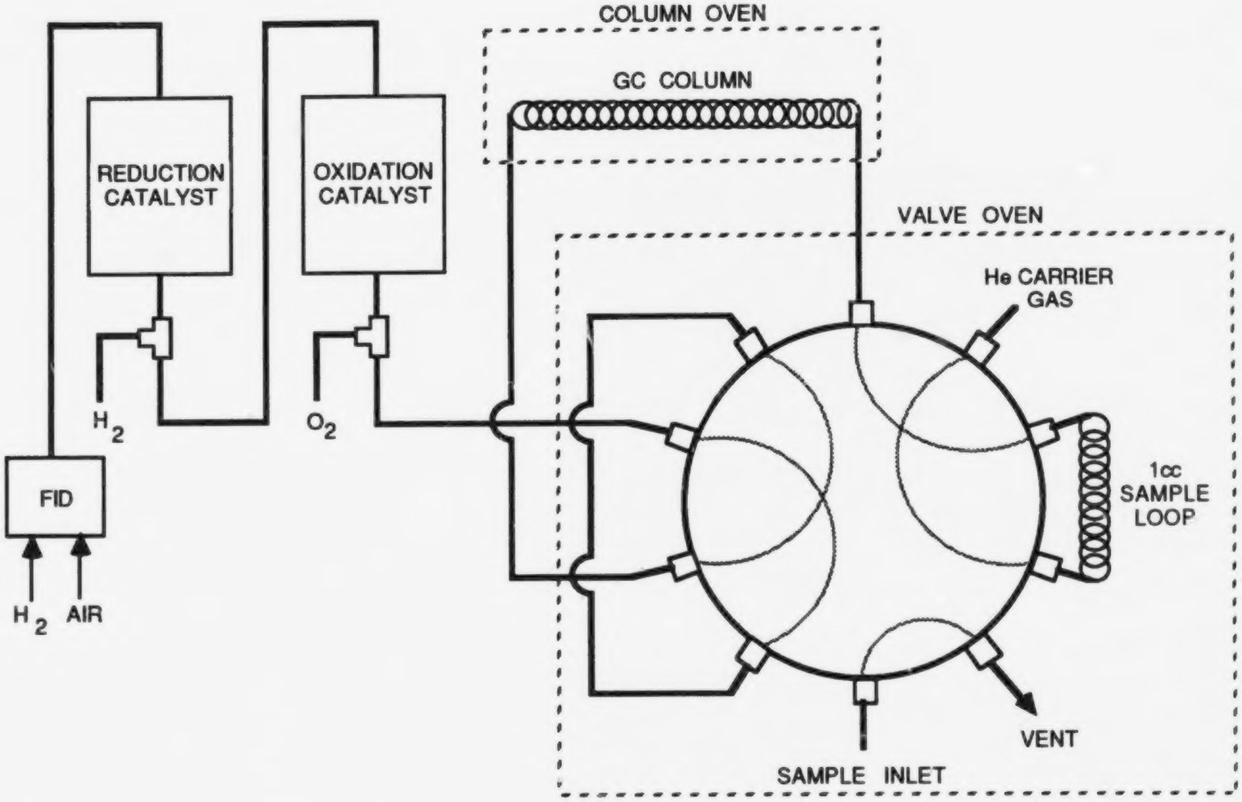
COLUMN OVEN
GC COLUMN
REDUCTION CATALYST
OXIDATION CATALYST
VALVE OVEN
He CARRIER GAS
H_2
O_2
FID
H_2
AIR
1cc SAMPLE LOOP
VENT
SAMPLE INLET

Figure 5.1. Nonmethane organic analyzer.

NMO Analyzer Catalyst Efficiency Testing

Date ______________________ Analyst ______________________

Oxidation Catalyst Temp., °C	Reduction Catalyst Temp., °C	FID Response			
		Run 1	Run 2	Run 3	Average

$$\text{Oxidation Catalyst Efficiency} = \frac{R_1 - R_2}{R_1} \times 100 \text{ (criteria is 99\% or greater)}$$

where: R_1 = Average FID response with both catalysts unheated.
R_2 = Average FID response with oxidation catalyst only heated.

$$\text{Reduction Catalyst Efficiency} = \frac{R_4}{R_3} \times 100 \text{ (criteria is 95\% or greater)}$$

where: R_3 = Average FID response with reduction catalyst only heated.
R_4 = Average FID response with both catalysts heated.

Figure 5.2. Analytical data form for NMO analyzer catalyst efficiency.

standards specified in Subsection 1.3.5 (i.e., 20 ppm, 200 ppm, and 3,000 ppm in air nominal). Convert certified concentrations in ppm to ppm C by multiplying ppm concentrations by 3. Record these concentrations on a data sheet, such as shown in Figure 5.3, along with the area responses observed in each injection. Calculate the mean response factor as ppm C/mean area for each standard and the overall mean response factor for all three standards. The NMO response linearity is acceptable if the average response factor of each calibration gas standard is within 2.5 percent of the overall mean response factor and if the relative standard deviation for each set of triplicate injections is less than 2 percent. If these criteria are not met, check the air and hydrogen flows for the FID to confirm that they are set according to manufacturer's specifications. Make adjustments if necessary and repeat the test. The overall mean response factor is used as the initial NMO calibration response factor (RF_{NMO}).

CO_2 Response Linearity Test and Initial Calibration - Perform the linearity test as described above, except use the CO_2 calibration standards specified in Subsection 1.3.5 (50 ppm, 500 ppm, and 1 percent in air). The overall mean response factor is used as the initial CO_2 calibration response factor (RF_{CO2}). The CO_2 calibration response factor (RF_{CO2}) should be within 10 percent of the NMO calibration factor (RF_{NMO}). If not, repeat the oxidation catalyst efficiency test.

NMO Analyzer Performance Test - After calibration of the NMO response as described above, analyze each of the test gas mixtures specified in Subsection 1.3.5 in triplicate. (Standard 1 is nominally 50 ppm CO, 50 ppm CH_4, 2 percent CO_2, and 20 ppm propane in air; Standard 2 is 50 ppm hexane in air; Standard 3 is nominally 20 ppm toluene in air; and Standard 4 is 100 ppm methanol in air.) Record the NMO area responses for each test gas on a data sheet such as shown in Figure 5.4. Convert the certified organic compound concentrations of the test gas mixtures to ppm C by multiplying by the carbon number of the compound (3 for propane, 6 for hexane, and 7 for toluene). Record these concentrations on the data sheet as the expected concentrations. Calculate the mean NMO concentration of the test gas using the equation shown in Figure 5.4. The analyzer performance is acceptable if the average measured NMO concentration for each mixture is within 5 percent of the expected value.

5.1.2 *Condensible Organic Recovery System*

Carrier Gas and Auxiliary Oxygen Blank Check - Each new tank of zero grade air and zero grade oxygen is analyzed with the NMO analyzer according to the procedure described in Subsection 5.4.2. The total concentration from any measured methane, carbon monoxide, carbon dioxide, or nonmethane organics should be less than 5 parts-per-million carbon (ppm C). Record data on a data sheet such as shown in Figure 5.5.

Oxidation Catalyst Efficiency Test - Perform this test using the equipment shown in Figure 5.6 and the following procedure:

1. Install a clean sample trap in the recovery system.
2. Replace the zero air carrier gas with the high level methane gas standard (1 percent methane in air nominal concentration).
3. Set the 4-port valve to the trap recovery position and the sample recovery valve to the vent position. Establish a 100 cm^3/minute flow of the methane in air standard.
4. Attach an intermediate collection vessel (ICV) to the recovery system.

NMO Analyzer Linearity Testing

Date ______________________ Analyst ______________________

Compound	Conc., ppm C	FID Area Response					RF, ppm C/Area	Overall Mean RF	Percent Difference
		Run 1	Run 2	Run 3	Mean	RSD			
Propane									
Propane									
Propane									
CO_2									
CO_2									
CO_2									

$$\text{Percent Difference} = \frac{\text{RF - Overall Mean RF}}{\text{Overall Mean RF}} \times 100$$

$$\text{RSD} = \frac{100}{\bar{R}} \quad \frac{(R_i - \bar{R})^2}{n - 1}$$

where: $\bar{R}$ = Mean RID response.
R_i = FID response for run i.
n = Number of runs.

$$\text{Mean RF} = \frac{\text{Standard Conc. (ppm C)}}{\text{Mean Area Response } (\bar{R})}$$

$$\begin{array}{l}\text{Overall Mean RF} \\ (RF_{NMO} \text{ or } RF_{CO2})\end{array} = \frac{RF_1 + RF_2 + RF_3}{3}$$

Figure 5.3. Analytical data form for NMO analyzer linearity tests.

NMO Analyzer Performance Testing

Date ______________________ Analyst ______________________

Test Gas	NMO Area Response				RF_{NMO}, ppm C/Area	Mean Conc., ppm C	Expected Conc., ppm C	Percent Diff.
	Run 1	Run 2	Run 3	Mean				
Propane Mix								
Hexane								
Toluene								
Methanol								

$$\text{Mean Conc.} = \text{Mean NMO Area} \times RF_{NMO}$$

$$\text{Percent Difference} = \frac{\text{Mean Conc.} - \text{Expected Conc.}}{\text{Expected Conc.}}$$

Figure 5.4. Analytical data form for NMO analyzer performance test.

Analysis of Recovery System Carrier Gases

Date ______________ Analyst ______________

Cylinder No.	Peak Area				RF_{NMO}	Concentration, ppm C				Total, ppm C
	CH_4	CO	CO_2	NMO		CH_4	CO	CO_2	NMO	

Concentration, ppm C = RF_{NMO} x Peak Area

Figure 5.5. Analytical data form for analysis of recovery system carrier gases.

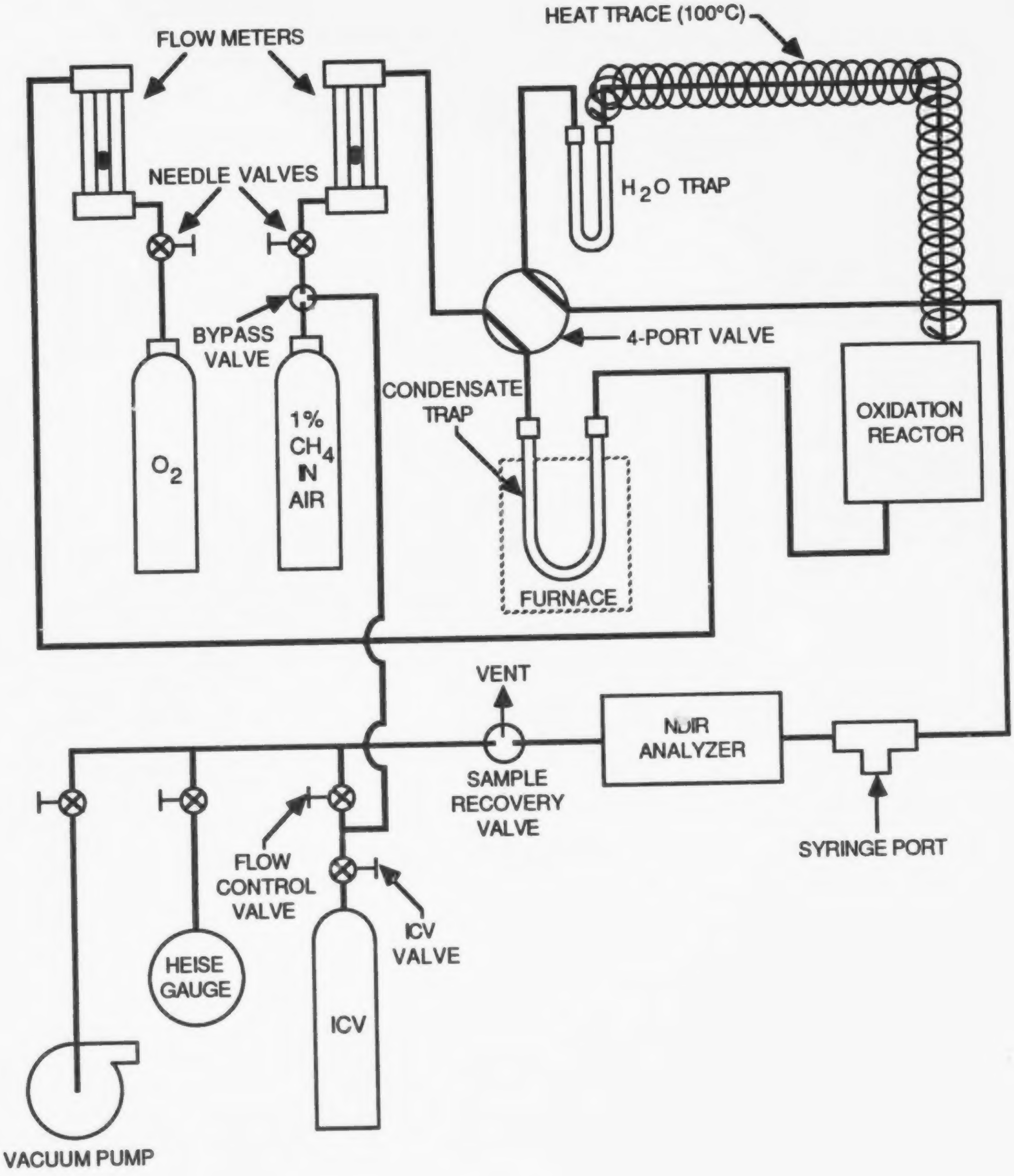
HEAT TRACE (100°C)
FLOW METERS
NEEDLE VALVES
H_2O TRAP
BYPASS VALVE
4-PORT VALVE
CONDENSATE TRAP
O_2
1% CH_4 IN AIR
OXIDATION REACTOR
FURNACE
VENT
NDIR ANALYZER
SAMPLE RECOVERY VALVE
SYRINGE PORT
FLOW CONTROL VALVE
ICV VALVE
HEISE GAUGE
ICV
VACUUM PUMP

Figure 5.6. Condensible organic recovery system oxidation catalyst efficiency test.

5. With the flow control and ICV valves fully open, open the vacuum valve to evacuate the manometer or gauge, connecting tubing, and the ICV to 10 mm Hg absolute pressure or less. Close the vacuum valve and flow control valve.
6. Once the NDIR analyzer response is stable, switch the sample recovery valve to the collect position. When the manometer or gauge begins to indicate pressure above atmospheric, open the flow control valve to maintain atmospheric pressure in the system. After fully opening the flow control valve, continue pressurizing the ICV to a nominal gauge pressure of 300 mm Hg (1060 mm Hg absolute).
7. Switch the sample recovery valve back to the vent position.
8. Close the ICV valve and detach the ICV from the system. Replace the methane standard with the zero air carrier gas.
9. Analyze the CO_2 concentration in the ICV using the NMO analyzer. The CO_2 concentration should be within 2 percent of the methane standard concentration. Record data on a from such as that shown in Figure 5.7. If the test criteria cannot be met, repack the oxidation catalyst tube with new material as described in Subsection 1.2.1.

Condensible Organic Recovery Efficiency Test - This test is performed using the equipment shown in Figure 5.8 and the following recommended procedure:

1. Install the liquid sample injection unit in place of a sample trap. Connect the end with the tee to the tubing terminated at the 4-port valve.
2. Set the zero air carrier gas and auxiliary oxygen flows to nominal levels of 100 cm^3/minute and 150 cm^3/minute, respectively.
3. Set the 4-port valve to the trap recovery position and the sample recovery valve to the vent position.
4. Attach an intermediate collection vessel to the recovery system. Evacuate the ICV, manometer or gauge, and connecting tubing to 10 mm Hg absolute pressure or less. Close the vacuum and flow control valves.
5. Switch the sample recovery valve to the collect position.
6. Inject 50 microliters of hexane into the septum port of the liquid sample injection unit.
7. Continue recovery of the injected organic as described in Subsection 5.3.2.
8. Record the final ICV volume and ICV pressure on a form such as that shown in Figure 5.9 and detach the ICV from the recovery system.
9. Determine the CO_2 concentration of the ICV by analysis using the NMO analyzer and record on a form such as that in Figure 5.9.
10. Calculate the percent recovery using the equation on a form such as that shown in Figure 5.9.
11. Repeat the recovery test two additional times with 50 ul hexane injections.
12. Perform additional recovery tests in triplicate with 10 ul hexane, 50 ul decane, and 10 ul decane each.

The recovery system performance is acceptable if the average percent recovery is 100 ± 10 percent with a relative standard deviation of less than 5 percent for each set of triplicate analyses. If these requirements are not met, check the recovery system for leaks and ensure adequate heating of the liquid sample injection unit during recovery.

Recovery System Oxidation Catalyst Testing

Date ____________________ Analyst ____________________

CO_2 Area Response of ICV	RF_{CO2} ppm/area	CO_2 Conc., ppm	CH_4 Std. Conc., ppm	Percent Difference

$$\text{Percent Difference} = \frac{CO_2\ \text{Conc.} - CH_4\ \text{Std. Conc.}}{CH_4\ \text{Std. Conc.}} \times 100$$

$$CO_2\ \text{Conc.} = CO_2\ \text{Area Response} \times RF_{CO2}$$

Figure 5.7. Analytical data form for recovery system oxidation catalyst testing.

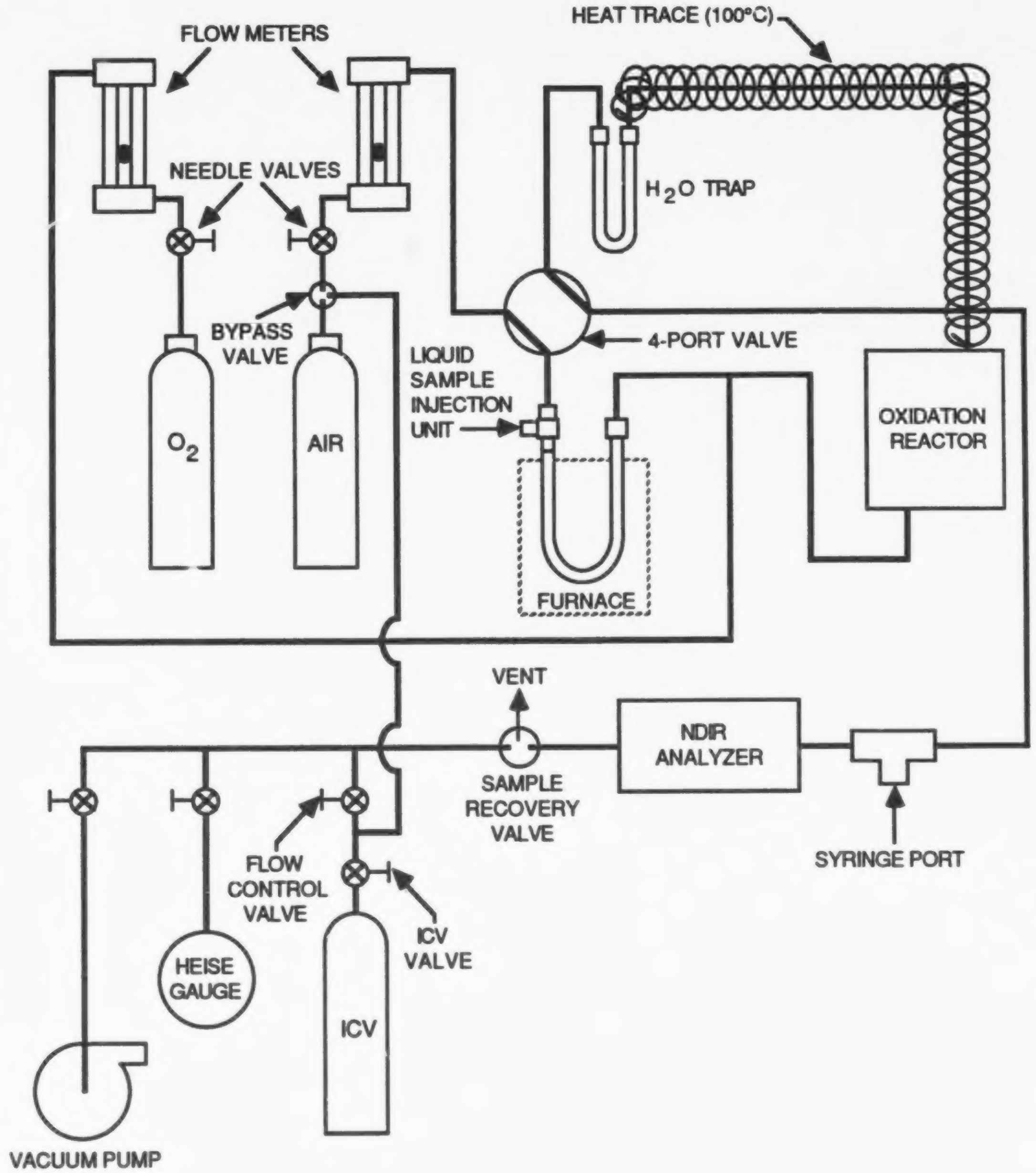
HEAT TRACE (100°C)
FLOW METERS
NEEDLE VALVES
H_2O TRAP
BYPASS VALVE
4-PORT VALVE
LIQUID SAMPLE INJECTION UNIT
O_2
AIR
OXIDATION REACTOR
FURNACE
VENT
NDIR ANALYZER
SAMPLE RECOVERY VALVE
SYRINGE PORT
FLOW CONTROL VALVE
ICV VALVE
HEISE GAUGE
ICV
VACUUM PUMP

Figure 5.8. Condensible organic recovery efficiency test.

Condensible Organic Recovery Efficiency Testing

Date ____________ Analyst ____________

Compound	Molecular Weight, g/g-mole	Density, g/ml	Volume Injected, uL	Collection Tank Data			CO_2 Conc., ppm	% Recovery	Overall Mean, % Recovery	RSD Percent
				Volume, m^3	Temp., °K	Pressure, mm Hg				
Hexane	86.18	0.661	10							
Hexane	86.18	0.661	10							
Hexane	86.18	0.661	10							
Hexane	86.18	0.661	50							
Hexane	86.18	0.661	50							
Hexane	86.18	0.661	50							
Decane	142.29	0.730	10							
Decane	142.29	0.730	10							
Decane	142.29	0.730	10							
Decane	142.29	0.730	50							
Decane	142.29	0.730	50							
Decane	142.29	0.730	50							

$$\text{Percent Recovery} = 1.604\,\frac{(\text{Molecular Weight})(\text{Tank Volume})(\text{Tank Pressure})(CO_2\ \text{Conc.})}{(\text{Volume Injected})(\text{Liquid Density})(\text{Tank Temp.})(\text{Carbon Number of Compound Injected})}$$

$$\text{RSD} = \frac{100}{\overline{\%R_i}}\sqrt{\frac{\Sigma(\%R_i - \overline{\%R_i})^2}{n - 1}}$$

where: $\%R_i$ = Percent recovery for an individual test.

$\overline{\%R_i}$ = Overall mean percent recovery for a compound and injection volume.

n = Number of tests (3).

Figure 5.9. Analytical data form for recovery efficiency test.

5.2 Daily Performance Tests

5.2.1 *Condensible Organic Recovery System* (see Figure 5.10) - Complete three tests each day before recovery of any samples.

Leak Test - Completely close the zero air and auxiliary oxygen metering valves and the flow control valve. Install a clean sample trap in the system and switch the sample recovery valve to the collect position. Open the vacuum valve and evacuate the system to 10 mm Hg absolute pressure or less. Close the vacuum valve and record the initial pressure on a data sheet such as shown in Figure 5.11. After 10 minutes, record the system pressure. The pressure change should be less than 2 mm Hg over the 10-minute period. If not, locate leakage by an appropriate method such as pressurizing and checking fittings with water. Repair leaks and retest as described above.

System Background Test - Set the carrier gas and auxiliary oxygen flows to their normal values of 100 cm^3/minute and 150 cm^3/minute, respectively. Switch the 4-port valve to the trap recovery position and the sample recovery valve to the vent position. Use a 10 cm^3 syringe to withdraw a sample from the syringe port located upstream of the NDIR analyzer. Flush the gas sampling loop on the NMO analyzer with the syringe sample and then switch the sampling valve to inject. Record the CO_2 peak area on a data sheet such as shown in Figure 5.12 and calculate the CO_2 concentration. The system background is acceptable if the CO_2 concentration measured is less than 10 ppm. If the background concentration is greater than 10 ppm, purge the recovery system with carrier gas and heat the trap connecting tubing to remove residual organics, then repeat the background test.

Oxidation Catalyst Efficiency Test - Conduct this test as described in Subsection 5.1.1. If the test criteria cannot be met, replace the oxidation catalyst and retest.

5.2.2 *NMO Analyzer Daily Calibration*

CO_2 Response Calibration - Analyze the highest level (1%) CO_2 calibration standard three times. Record the CO_2 peak areas on a data sheet such as that shown in Figure 5.13 and calculate the average daily response factor (DRF_{CO2}). The average daily response factor (DRF_{CO2}) should be within 5 percent of the initial CO_2 calibration response factor (RF_{CO2}). If not, repeat the initial performance test as described in Subsection 5.1.2 to establish a new RF_{CO2}. The daily response factor is used to quantitate the CO_2 concentrations of the ICV samples.

NMO Response Calibration - Analyze the gas mixture containing nominal concentrations of 50 ppm CO, 50 ppm CH_4, 2 percent CO_2, and 20 ppm propane in air in triplicate. Record the NMO peak areas on a data sheet such as that shown in Figure 5.13 and calculate the average daily response factor (DRF_{NMO}). The average daily NMO response factor (DRF_{NMO}) should be within 5 percent of the initial NMO response factor (RF_{NMO}). If not, repeat the initial performance test as described in Subsection 5.1.2 to establish a new RF_{NMO}. The daily response factor is used to quantitate the NMO concentration of the sample tanks.

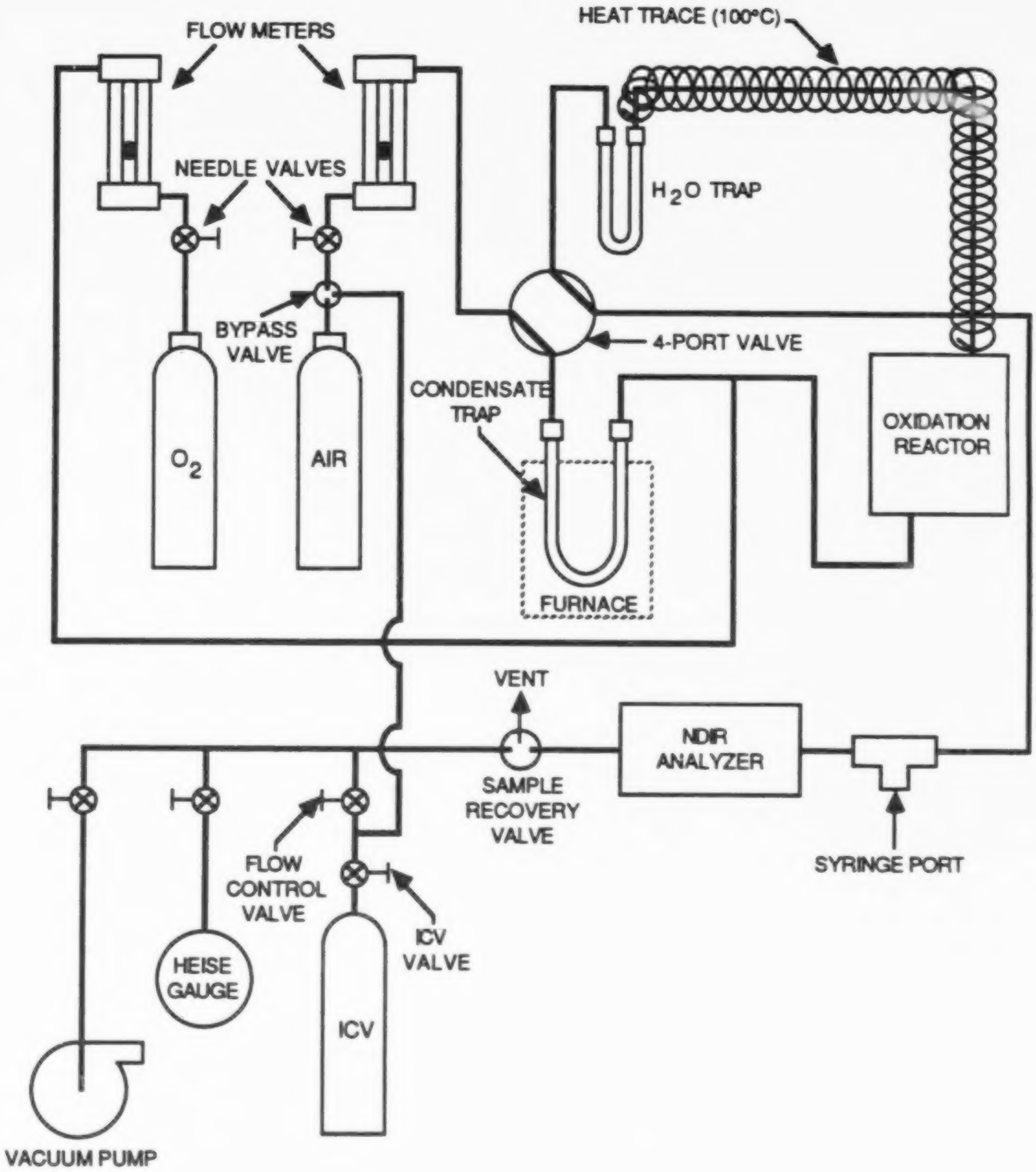
HEAT TRACE (100°C)
FLOW METERS
NEEDLE VALVES
H_2O TRAP
BYPASS VALVE
4-PORT VALVE
CONDENSATE TRAP
O_2
AIR
OXIDATION REACTOR
FURNACE
VENT
NDIR ANALYZER
SAMPLE RECOVERY VALVE
SYRINGE PORT
FLOW CONTROL VALVE
ICV VALVE
HEISE GAUGE
ICV
VACUUM PUMP

Figure 5.10. Condensate recovery system.

Condensate Recovery System Leak Testing

Date	Analyst	Initial Time	Initial Press., Torr	Final Time	Final Press., Torr

Figure 5.11. Analytical data form for recovery system leak test.

Condensate Recovery System Background Testing

Date	Analyst	RF_{CO2}, ppm/Area	CO_2 Peak Area	CO_2 Conc., ppm

CO_2 conc., ppm = CO_2 Peak Area x RF_{CO2}

Figure 5.12. Analytical data form for recovery system background test.

Daily NMO Analyzer Calibration

Date ____________________ Analyst ______________________________

NMO Calibration Cylinder No. __________ CO_2 Calibration Cylinder No. ______

Compound	Conc., ppm C	FID Area Response				DRF, ppm C/Area	Initial RF	Percent Diff.
		Run 1	Run 2	Run 3	Mean			

$$\text{Percent Difference} = \frac{\text{DRF} - \text{Initial RF}}{\text{Initial RF}} \times 100$$

$$\text{Mean Area Response} = \frac{R_1 + R_2 + R_3}{3}$$

where: R_i = FID area response.

$$\text{DRF} = \frac{\text{Concentration}}{\text{Mean Area Concentration}}$$

Figure 5.13. Analytical data form for daily calibration of NMO analyzer.

5.3 Condensible Organic Fraction Recovery

Recovery of condensible organics is accomplished in two steps. First, the condensate trap is purged of carbon dioxide while cooling the trap in dry ice. Second, the condensible organics are volatilized and converted catalytically to carbon dioxide which is collected in an intermediate collection vessel (ICV) for analysis.

5.3.1 *Trap Purge and Sample Tank Pressurization* - The following procedure is used to purge carbon dioxide from the condensate trap and to pressurize the sample tank (see Figure 5.14):

1. Obtain the sample tank and condensate trap from the source test run to be analyzed (or the blank sample tank and blank condenste trap).
2. Set the zero air carrier gas to a flow rate of 100 cm^3/minute and the auxiliary oxygen flow to zero.
3. Switch the 4-port valve to the CO_2 purge position.
4. Attach the sample tank to the condensate trap recovery system.
5. With the sample recovery valve in the vent position and the flow control valve fully open, evacuate the manometer or gauge to the expected pressure of the sample tank.
6. Close the vacuum valve, open the sample tank valve, and record the sample tank pressure (P_t) in mm Hg absolute on a data sheet such as that shown in Figure 5.15.
7. Immerse the condensate trap in crushed dry ice and attach to the recovery system with the trap outlet connected to the tube terminating at the 4-port valve.
8. Switch the sample recovery valve from vent to collect. Adjust the flow control valve to maintain approximately atmospheric pressure in the recovery system.
9. After the NDIR analyzer responds to the CO_2 purged from the trap and the response reaches a minimum level, withdraw a 10 cm^3 syringe sample from the syringe port and analyze with the NMO analyzer. Repeat analyses until the CO_2 concentration of the trap effluent is less than 5 ppm. The length of time required to purge the trap of residual CO_2 will depend upon the internal volumes of the condensate trap recovery system. A larger volume system will require more purging time at the specified flow rates to meet the effluent concentration criteria of less than 5 ppm.
10. Switch the carrier gas bypass valve to pressurize the sample tank to approximately 1060 mm Hg absolute pressure. Switch the sample recovery valve to the vent position and record the final sample tank pressure (P_{tf}) on the data sheet. Detach the sample tank from the system.

5.3.2 *Recovery of Condensible Organics* - The following procedure is used to purge organics from the sample trap, convert them to carbon dioxide, and collect the carbon dioxide in an intermediate collection vessel (see Figure 5.16).

1. Attach an ICV to the trap recovery system. Open the flow control and ICV values fully and evacuate the manometer or gauge, connecting tube, and ICV to 10 mm Hg absolute pressure or less. Close the flow control and vacuum valves.
2. Set the auxiliary oxygen flow to a rate of 150 cm^3/minute.
3. Switch the 4-port valve to the trap recovery position and the sample recovery valve to the collect position. After the system reaches atmospheric pressure, adjust the flow control valve to maintain atmospheric pressure within 10 percent.

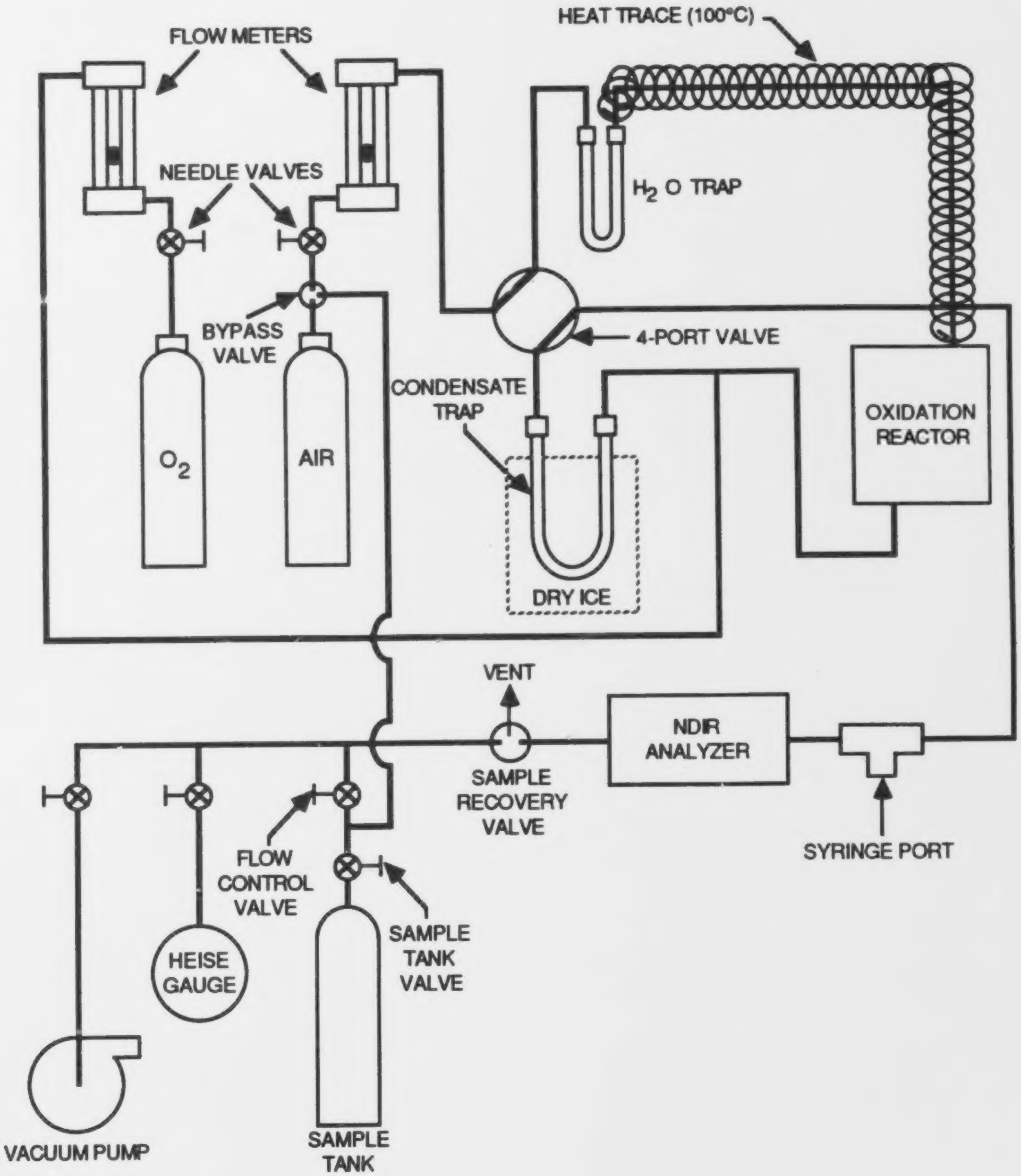
HEAT TRACE (100°C)
FLOW METERS
NEEDLE VALVES
H_2O TRAP
BYPASS VALVE
4-PORT VALVE
CONDENSATE TRAP
O_2
AIR
OXIDATION REACTOR
DRY ICE
VENT
NDIR ANALYZER
SAMPLE RECOVERY VALVE
SYRINGE PORT
FLOW CONTROL VALVE
HEISE GAUGE
SAMPLE TANK VALVE
VACUUM PUMP
SAMPLE TANK

Figure 5.14. Condensate recovery system, CO_2 purge.

Date ________ Analyst ______________________ Plant ______________________
Location ______________________________ Date Sampled ______________________
Run No. ________________ Trap No. ______________________________
Sample Tank No. __________________________ ICV No. ______________________

ICV volume, m^3 (V_v) ________
Sample tank pressure after sampling, mm Hg (P_t) ________
Sample tank pressure after pressurizing, mm Hg (P_{tf}) ________
ICV final pressure, mm Hg (P_f) ________
ICV volume, m^3 (V_v) ________
Sample tank temperature after pressurizing, °K (T_{tf}) ________
ICV final temperature, °K (T_f) ________
Sample tank temperature at end of sampling, °K (T_t) ________
Sample tank temperature before sampling, °K (T_{ti}) ________
Sample tank pressure before sampling, mm Hg (P_{ti}) ________
Gas volume sampled, dsm^3 (V_s) ________

	ICV Analysis				Sample Tank Analysis	
Run No.	CO_2 Area	CO_2 Conc., ppm (C_{cm1})	NMO Area	NMO Conc., ppm C (C_{cm2})	NMO Area	NMO Conc., ppm C (C_{tm})
1						
2						
3						
Mean						

CO_2 Conc. = CO_2 Area x DRF_{CO_2}

NMO Conc. = NMO Area x DRF_{NMO}

Noncondensible Organic Concentration, ppm C (C_t) $= \dfrac{\dfrac{P_{tf}}{T_{tf}}}{\dfrac{P_t}{T_t} - \dfrac{P_{ti}}{T_{ti}}} \times C_{tm}$

Condensible Organic Concentration, ppm C (C_c) $= 0.3857 \dfrac{V_v\ P_f}{V_s\ T_f} \times (C_{cm_1} + C_{cm_2})$

TGNMO Concentration, ppm C = $C_t - C_{tb} + C_c - C_{cb}$*

*Note: Blank subtraction must have prior approval of the Administrator.

Figure 5.15. Analytical data form for sample recovery and analysis.

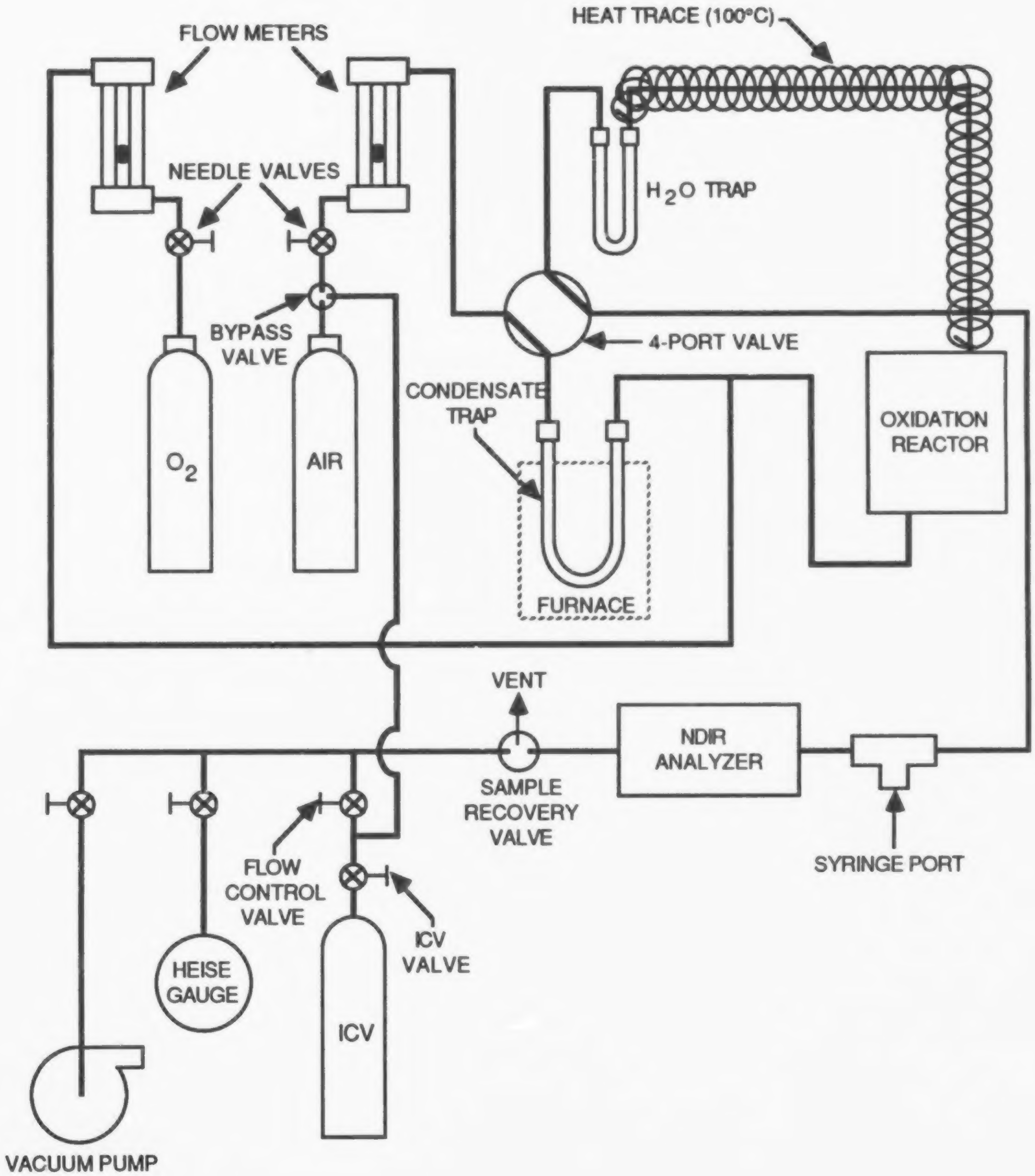
FLOW METERS
HEAT TRACE (100°C)
NEEDLE VALVES
H_2O TRAP
BYPASS VALVE
4-PORT VALVE
CONDENSATE TRAP
O_2
AIR
OXIDATION REACTOR
FURNACE
VENT
NDIR ANALYZER
SAMPLE RECOVERY VALVE
SYRINGE PORT
FLOW CONTROL VALVE
ICV VALVE
HEISE GAUGE
ICV
VACUUM PUMP

Figure 5.16. Condensate recovery system, collection of trap organics.

4. Remove the condensate trap from the dry ice and allow ambient air warming of the trap while monitoring the NDIR analyzer response. The auxiliary oxygen flow may be discontinued after five minutes if the CO_2 response is below 10,000 ppm (1%).
5. Heat the trap by placing it in a furnace at 200°C. If the NDIR response exceeds 50,000 ppm (5%) during recovery, resume auxiliary oxygen flow at a rate of 150 cm^3/minute.
6. After the NDIR analyzer indicates a CO_2 concentration of less than 10,000 ppm, begin heating the tubing connecting the condensate trap to the oxidation catalyst with a heat gun. Heat the tubing slowly along the entire length from the trap to the catalyst, and repeat two additional times.
7. Continue trap heating and purging until the CO_2 concentration is below 10 ppm (determined by analyzing syringe samples collected before NDIR analyzer with the NMO analyzer).
8. When recovery is complete, switch the carrier gas bypass to pressurize the ICV to approximately 1060 mm Hg. Switch the sample recovery valve to vent and record the ICV final pressure (P_f) on a data sheet such as that shown in Figure 5.15.

5.4 Analysis

The total source concentration of gaseous nonmethane organics is determined by combining the noncondensible and condensible concentrations. The noncondensible concentration is determined by analyzing the sample tank for nonmethane organics, and the condensible concentration is determined by analyzing the intermediate collection vessel for CO_2.

5.4.1 *NMO Analyzer Operating Conditions* - Set the helium carrier gas flow rate to 30 cm^3/minute. Set the oxidation catalyst oxygen flow rate to 2.2 cm^3/minute. Heat the column oven to an initial temperature of 85°C.

5.4.2 *Intermediate Collection Vessel Analysis* - Analyze the ICV contents as follows:

1. Attach the ICV to the 10-port gas sampling valve.
2. Purge the sample loop with gas from the ICV and then switch the 10-port valve to the inject position.
3. When the detector response returns to near baseline following the CO_2 peak, switch the 10-port valve to the backflush position and increase the column oven temperature to 195°C as rapidly as possible.
4. After detection of any nonmethane organic compounds, return the column oven temperature to 85°C.
5. Record the CO_2 peak area and NMO peak area on the data sheet shown in Figure 5.15.
6. Repeat the analysis two additional times.
7. Calculate CO_2 and NMO concentrations using equations given in Figure 5.15.
8. Calculate the average CO_2 concentration (C_{cm1}) and the average NMO concentration (C_{cm2}) in the ICV and record on the data sheet.

5.4.3 *Sample Tank Analysis* - Analyze the sample tank as described in Section 5.4.2. Record the NMO peak area only and calculate the average NMO concentration (C_{tm}) of the sample tank.

5.4.4 *Condensible Organic Blank Analysis* -

1. Analyze the ICV resulting from the blank trap recovery as described in Section 5.4.2.
2. Calculate CO_2 and NMO concentrations using equations given in Figure 5.16.
3. Calculate the average CO_2 concentration (C_{cm1}) and the average NMO concentration (C_{cm2}) in the blank ICV and record on the data sheet.
4. Calculate the source condensible organic blank concentration (C_{cb}) using the equation in Figure 5.17. The condensible organic blank concentration may not exceed 15 ppmC. If the blank value exceeds 15 ppmC, then the value of 15 ppmC may be used as the blank value. NOTE: The method does not provide for blank correction. However, with prior approval of the Administrator, blank correction (subtracting the blank value) may be used.

5.4.5 *Noncondensible Organic Blank Analysis* - Analyze the blank sample tank as described in Section 5.4.2. Record the NMO peak area only and calculate the average NMO concentration (C_{tmb})of the blank sample tank. Calculate the source noncondensible organic blank concentration (C_{tb}) using the equation shown in Figure 5.17. The noncondensible organic blank concentration may not exceed 5 ppmC. If the blank value exceeds 5 ppmC, then the value of 5 ppmC may be used as the blank value. NOTE: The method does not provide for blank correction. However, with prior approval of the Administrator, blank correction (subtracting the blank value) may be used.

5.4.6 *Source Concentration Calculations* - Calculate the noncondensible organic concentration (C_t), the condensible organic concentration (C_c), and the TGNMO concentration using the data and equations shown Figures 5.15 and 5.17 (blank subtraction requires prior approval of Adminstrator).

Date ________ Analyst ______________________________ Plant ______________________________
Location ____ Date Sampled ______________________________
Blank No. ____________________ Trap No. ____
Sample Tank No. ________________________________ ICV No. ______________________________

ICV volume, m^3 (V_v) ________
Sample tank pressure after sampling, mm Hg (P_t) ________
Sample tank pressure after pressurizing, mm Hg (P_{tf}) ________
ICV final pressure, mm Hg (P_f) ________
ICV volume, m^3 (V_v) ________
Sample tank temperature after pressurizing, °K (T_{tf}) ________
ICV final temperature, °K (T_f) ________
Sample tank temperature at end of sampling, °K (T_t) ________
Sample tank temperature before sampling, °K (T_{ti}) ________
Sample tank pressure before sampling, mm Hg (P_{ti}) ________
Gas volume sampled, dsm^3 (V_s) ________

	ICV Analysis				Sample Tank Analysis	
Blank No.	CO_2 Area	CO_2 Conc., ppm (C_{cm1})	NMO Area	NMO Conc., ppm C (C_{cm2})	NMO Area	NMO Conc., ppm C (C_{tm})
1 2 3						
Mean						

CO_2 Conc. = CO_2 Area x DRF_{CO_2}

NMO Conc. = NMO Area x DRF_{NMO}

Noncondensible Organic Blank Concent, ppm C (C_{tb}) =
$$\frac{\dfrac{P_{tf}}{T_{tf}}}{\dfrac{P_t}{T_t} - \dfrac{P_{ti}}{T_{ti}}} \times C_{tm}$$

Condensible Organic Blank Concent, ppm C (C_{cb}) =
$$0.3857 \, \frac{V_v \, P_f}{V_s \, T_f} \times (C_{cm_1} + C_{cm_2})$$

Figure 5.17. Analytical data form for blank sample recovery and analysis.

Date _____ Plant Name __________________ Sampling Location ________________

Initial Performance Tests of Condensible Organic Recovery System

Do zero grade air and zero grade oxygen carrier gases contain less than 5 ppm C total of methane, carbon monoxide, carbon dioxide, and nonmethane organics? ___ yes ___ no. (If no, replace gases or filter to remove impurities.)

Does the CO_2 concentration in the ICV collected during the oxidation catalyst efficiency test agree within 2 percent of the methane standard concentration? ___ yes ___ no. (If no, replace the oxidation catalyst.)

Is recovery efficiency 100 ± 10 percent with a relative standard deviation of less than 5 percent for each set of triplicate injections of hexane and decane at 10 ul and 50 ul? ____ yes ____ no. (If no, check the recovery system for leaks and assure adequate heating of the liquid sample injection unit.)

Initial Performance Tests of NMO Analyzer

Does the oxidation catalyst efficiency test show an efficiency of 99 percent or better? ____ yes ____ no. (If no, replace the oxidation catalyst.)

Does the reduction catalyst efficiency test show an efficiency of 95 percent or better? ____ yes ____ no. (If no, replace the reduction catalyst.)

Is the NMO response linear? (Average response factor of each calibration standard within 2.5 percent of the overall mean and a relative standard deviation for each set of triplicate injections of less than 2 percent.) ____ yes ____ no. (If no, check FID air and hydrogen flows and make adjustments.)

Is the CO_2 response linear? (Average response factor of each calibration standard within 2.5 percent of the overall mean and a relative standard deviation for each set of triplicate injections of less than 2 percent). ____ yes ____ no. (If no, check FID air and hydrogen flows and make adjustments.)

Are the measured concentrations of the analyzer performance test gases within 5 percent of their certified concentrations? ____ yes ____ no. (If no, replace the GC column or adjust column backflush timing and/or temperature to obtain acceptable performance.)

Daily Performance Tests of Condensible Organic Recovery System

Leak test completed? ____ yes ____ no. (Leak test following procedure in Subsection 5.2.1.)

System background test completed? ____ yes ____ no. (Follow procedure in Subsection 5.2.1.)

Oxidation catalyst efficiency test completed? ____ yes ____ no. (Follow procedure in Subsection 5.2.1.)

Daily Calibration of NMO Analyzer

Calibration of CO_2 response completed with 1% CO_2 in air standard? ____ yes ____ no.

Calibration of NMO response completed with mixed gas standard containing 50 ppm CO, 50 ppm CH_4, 2 percent CO_2, and 20 ppm propane in air? ____ yes ____ no.

Figure 5.18. Postsampling operations checklist.

(Continued)

Figure 5.18. (Continued)

Condensible Organic Recovery

Condensate trap purged and sample tank pressurized? ____ yes ____ no. (Follow procedure in Subsection 5.3.1.)

Condensible organics purged from sample trap, converted to CO_2, and collected in an ICV? ____ yes ____ no. (Follow procedure in Subsection 5.3.2.)

Analysis of Sample Tank and Intermediate Collection Vessel

NMO analyzer operating conditions set? ____ yes ____ no. See Subsection 5.4.1.)

ICV analyzed in triplicate? ____ yes ____ no. (Follow procedure in Subsection 5.4.2.)

Sample tank analyzed in triplicate? ____ yes ____ no. (Follow procedure in Subsection 5.4.3.)

Source concentration calculated? ____ yes ____ no. (Use equations shown in Figure 5.1.5.)

Table 5.1. ACTIVITY MATRIX FOR SAMPLE ANALYSIS

Characteristic	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Carrier gas and auxiliary O_2 used in condensible organic recovery system	Total concentration from CH_4, CO, CO_2, and NMO must be <5 ppm C	Analyze each new cylinder with the NMO analyzer	Obtain better grades of gas from vendor or filter gases to reduce impurities
Recovery system oxidation catalyst efficiency	CO_2 concentration of collected sample must be ± 2% of CH_4 test gas concentration	Before first operation after any shutdown of longer than 6 months, after modification, or daily when samples are analyzed; test by replacing carrier gas with 1% CH_4 in air, collection of sample in ICV, and analysis with NMO analyzer	Replace oxidation catalyst
Condensible organic recovery efficiency	Average recovery of 100 ± 10% with a RSD of <5% for each set of triplicate analyses	Before first operation after any shutdown of longer than 6 months, or after modification; test with 10 and 50 ul injections of hexane and decane into liquid sample injection unit and analysis of collected sample with NMO analyzer	Check recovery system for leaks and assure adequate heating of liquid sample injection unit during recovery
NMO analyzer oxidation catalyst efficiency	FID response with oxidation catalyst heated must be <1% of response with both catalysts unheated	Before first operation after any shutdown of longer than 6 months, or after modification; analyze 1% CH_4 in air with both catalysts unheated and then with oxidation catalyst only heated	Replace oxidation catalyst

(Continued)

Table 5.1 (Continued)

Characteristic	Acceptance Limits	Frequency and method of measurement	Action if requirements are not met
NMO analyzer reduction catalyst efficiency	Analyzer response to 1% methane in air standard with both catalysts heated should agree ± 5% of response, with reduction catalyst only heated	Before first operation after any shutdown of more than 6 months, or after modification; a 1% CH_4 in air standard is analyzed with both catalysts heated and with the reduction catalyst only heated	Replace reduction catalyst
NMO response linearity and calibration	Response factor of each calibration gas standard must be ±2.5% of overall mean response factor and relative standard deviation of each set of triplicate analyses must be <2%	Before first operation after any shutdown of longer than 6 months, or after modification; analyze propane in air standards with NMO analyzer under normal operating conditions	Check air and hydrogen flows for FID to confirm that they are set according to manufacturer's specifications; make adjustments if necessary and and repeat test
CO_2 response linearity and calibration	Response factor of each calibration gas standard must be ±2.5% of overall mean response factor and relative standard deviation of each set of triplicate analyses must be < 2%	Before first operation after any shutdown of longer than 6 months, or after modification; analyze the CO_2 standards with NMO analyzer under normal operating conditions	Check air and hydrogen flows for FID to confirm they are set according to manufacturer's specifications; make adjustments if necessary and repeat test
NMO analyzer performance	Average concentration based on triplicate analysis must be within 5% of expected value for each test mixture	Before first operation after any shutdown of longer than 6 months, or after modification; analyze test mixtures with NMO anlayzer under normal operating conditions	Replace GC column or adjust column backflush timing and/or temperature to obtain acceptable performance

(continued)

TABLE 5.1 (continued)

Characteristic	Acceptance Limits	Frequency and method of measurement	Action if requirements are not met
Condensible organic recovery system leak test	System should lose <2 mm Hg vacuum over a 10-min period	Daily, before analysis of samples; evacuate the recovery system and monitor the vacuum with mercury manometer or pressure gauge	Locate leakage by appropriate method such as pressurizing and checking fittings with water; repair leaks and retest
Recovery system background test	The measured CO_2 background concentration must be <10 ppm	Daily, before analysis of samples; analyze syringe samples of recovery system effluent with NMO analyzer	Purge recovery system with carrier gas and heat trap connecting tubing to remove residual organics, then retest
CO_2 response calibration	The average response factor from triplicate analysis of highest concentration standard must be ±5% of initial CO_2 response factor	Before and after analysis of each set of samples or daily, whichever occurs first, analyze highest level CO_2 standard with NMO analyzer under normal operating conditions	Repeat analyzer catalyst efficiency tests, linearity tests, and performance test
NMO response calibration	Average NMO response factor should be ±5% of initial NMO response factor	Before and after analysis of each set of samples or daily, whichever occurs first, analyze gas mixture containing 50 ppm CO, 50 ppm CH_4, 2% CO_2, and 20 ppm propane in air	Repeat analyzer catalyst efficiency tests, linearity tests, and performance test
Sample trap purge and sample tank pressurization	CO_2 concentration of syringe port samples must be less than 5 ppm	Analyze syringe port samples with NMO analyzer after NDIR analyzer response returns to baseline	Continue purging trap with carrier gas and analyze additional syringe port samples

(Continued)

TABLE 5.1 (continued)

Characteristic	Acceptance Limits	Frequency and method of measurement	Action if requirements are not met
Condensible organics recovery	CO_2 concentration of syringe port samples must be <10 ppm	Analyze syringe port samples with NMO analyzer when NDIR analyzer response returns to baseline	Continue heating of sample trap and purging with carrier gas
Intermediate collection vessel analysis	Relative standard deviation from triplicate analysis for CO_2 and NMO must be <2%	Analyze ICV after trap recovery by injecting aliquots on NMO analyzer	Perform additional analyses until RSD of last three injections is <2%
Sample tank analysis	Relative standard deviation from triplicate analysis for NMO must be <2%	Analyze sample tank after trap purging and tank pressurization by injecting aliquots on NMO analyzer	Perform additional analyses until RSD of last three injections is <2%

6.0 CALCULATIONS

Calculation errors due to procedural or mathematical mistakes can be a part of total system error. Therefore, it is recommended that each set of calculations be repeated or spotchecked, preferably by a team member other than the one who performed the original calculations. If a difference greater than typical round-off error is detected, the calculations should be checked step-by-step until the source of error is found and corrected.

Calculations should be carried out to at least one extra decimal figure beyond that of the acquired data and should be rounded off after final calculation to two significant digits for each run or sample. All rounding of numbers should be performed in accordance with the ASTM 380-76 procedures. All calculations should then be recorded on a calculation form such as Figure 6.1.

A computer program is advantageous in reducing calculation errors. If a computer program is used, the original data entered should be included in the printout so it can be reviewed; if differences are observed, a new computer run should be made. A computer program is also useful in maintaining a standardized format for reporting of results. It is highly recommended that a standardized format including the data shown in Figure 6.2 be used for reporting the emissions results. The data shown will allow auditing of the calculations.

Table 6.1 at the end of this section summarizes the quality assurance activities for calculations.

6.1 Nomenclature

The following nomenclature is used in the calculations:

C = TGNMO concentration of the effluent, ppm C equivalent.
C_c = Calculated condensible organic (condensate trap) concentration of the effluent, ppm C equivalent.
C_{cb} = Calculated condensible organic (condensate trap) blank concentration of the sampling equipment, ppm C equivalent.
C_{cm} = Measured concentration (NMO analyzer) for the condensate trap ICV, ppm CO_2.
C_{cmb} = Measured blank concentration (NMO analyzer) for the condensate trap ICV, ppm CO_2.
C_t = Calculated noncondensible organic concentration (sample tank) of the effluent, ppm C equivalent.
C_{tb} = Calculated noncondensible organic blank concentration (sample tank) of the sampling equipment, ppm C equivalent.
C_{tm} = Measured concentration (NMO analyzer) for the sample tank, ppm NMO.
C_{tm} = Measured blank concentration (NMO analyzer) for the sample tank, ppm NMC.
F = Sampling flow rate, cc/min.
L = Volume of liquid injected, ul.
M = Molecular weight of the liquid injected, g/g-mole.
m_c = TGNMO mass concentration of the effluent, mg C/dsm^3.

N = Carbon number of the liquid compound injected ($N = 12$ for decane, $N = 6$ for hexane).
P_f = Final pressure of the intermediate collection vessel, mm Hg absolute.
P_b = Barometric pressure, cm Hg.
P_{ti} = Gas sample tank pressure before sampling, mm Hg absolute.
P_t = Gas sample tank pressure after sampling, but before pressurizing, mm Hg absolute.
P_{tf} = Final gas sample tank pressure after pressurizing, mm Hg absolute.
T_f = Final temperature of intermediate collection vessel, °K.
T_{ti} = Sample tank temperature before sampling, °K.
T_t = Sample tank temperature at completion of sampling, °K.
T_{tf} = Sample tank temperature after pressurizing, °K.
V = Sample tank volume, m^3.
V_t = Sample train volume, cc.
V_v = Intermediate collection vessel volume, m^3.
V_s = Gas volume sampled, dsm^3.
n = Number of data points.
q = Total number of analyzer injections of intermediate collection vessel during analysis (where k = injection number, $1 \ldots q$).
r = Total number of analyzer injections of sample tank during analysis (where j = injection number, $1 \ldots r$).
x_i = Individual measurements.
x = Mean value.
ρ = Density of liquid injected, g/cc.
θ = Leak check period, min.
ΔP = Allowable pressure change, cm Hg.

6.2 Calculations

The following are the equations used with the example calculation form, Figure 6.1 to calculate the concentration of TGNMO, the allowable limit for the pretest leak check, and assess the efficiency of the condensate recovery system.

6.2.1 *Allowable Pressure Change* - Calculate the allowable pressure change, in cm Hg, for the pretest leak check using the following equation. This value is then compared to the actual pressure change, in cm Hg, to determine if the train is suitable for sampling.

$$\Delta P = 0.01 \frac{F P_b \theta}{V_t} \qquad \text{Equation 6-1}$$

6.2.2 *Sample Volume* - For each test run, calculate the gas volume sampled using the following equation.

$$V_s = 0.3857\, V \left[\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}} \right] \qquad \text{Equation 6-2}$$

6.2.3 *Noncondensible Organics Concentration* - For each sample tank, determine the concentration of nonmethane organics, in ppm C, using Equation 6-3.

6.2.3 *Noncondensible Organics Concentration* - For each sample tank, determine the concentration of nonmethane organics, in ppm C, using Equation 6-3.

$$C_t = \left[\frac{\dfrac{P_{tf}}{T_{tf}}}{\dfrac{P_t}{T_t} - \dfrac{P_{ti}}{T_{ti}}}\right]\left[\frac{1}{r}\sum_{j=1}^{r} C_{tm_j}\right] \qquad \text{Equation 6-3}$$

6.2.4 *Noncondensible Organics Blank Concentration* - For blank sample tank, determine the concentration of nonmethane organics, in ppm C, using Equation 6-3 and the values for C_{tmb}. The blank value may not exceed 5 ppm. If the blank value exceeds 5 ppm C, then the value of 5 ppm C may be used as the blank value. The calculated blank value is C_{tb}.

6.2.5 *Condensible Organics Concentration* - For each condensate trap, determine the concentration of organics, in ppm C, using Equation 6-4.

$$C_c = 0.3857\ \frac{V_v\ P_f}{V_s\ T_f}\left[\frac{1}{q}\sum_{k=1}^{q} C_{cm_k}\right] \qquad \text{Equation 6-4}$$

6.2.6 *Condensible Organics Concentration* - For each condensate trap, determine the concentration of organics, in ppm C, using Equation 6-4 and the values for C_{cmb}. The blank value may not exceed 15 ppm. If the blank value exceeds 15 ppm C, then the value of 15 ppm C may be used as the blank value. The calculated blank value is C_{cb}.

6.2.7 *TGNMO Concentration* - To determine the TGNMO concentration for each test run, use Equation 6-5. NOTE: The method does not provide for blank correction. The tester must have prior approval of the Administrator to use blank correction (subtract blank).

$$C = C_t - C_{tb} + C_c - C_{cb} \qquad \text{Equation 6-5}$$

6.2.8 *TGNMO Mass Concentration* - To determine the TGNMO mass concentration as carbon for each test run, use Equation 6-6.

$$m_c = 0.4993\ C \qquad \text{Equation 6-6}$$

6.2.9 *Percent Recovery* - Calculate the percent recovery for the liquid organic injections used to assess the efficiency of the condensate recovery and conditioning system using Equation 6-7. The average recovery for triplicate injections shoul fall within 10% (of 100%).

$$\text{Percent Recovery} = 1.604 \frac{M}{L} \frac{V_v}{\rho} \frac{P_f}{T_f} \frac{C_{cm}}{N} \qquad \text{Equation 6-7}$$

6.2.10 *Relative Standard Deviation* - Calculate the relative standard deviation (RSD) for the percent recoveries for triplicate injections of liquid organics using Equation 6-8. The RSD should be less than 5% for each set of triplicate analyses.

$$RSD = \frac{100}{\bar{x}} \sqrt{\frac{\Sigma(x_i - \bar{x})^2}{n - 1}} \qquad \text{Equation 6-8}$$

ALLOWABLE PRETEST LEAK CHECK PRESSURE CHANGE

F = __ __ cc/min, P_b = __ __ . __ __ cm Hg, θ = __ __ . __ min,

V_t = __ __ __ __ cc

$$\Delta P = 0.01 \frac{FP_b \theta}{V_t} = \_ . \_\_ \text{ cm Hg} \qquad \text{Equation 6-1}$$

SAMPLE VOLUME

V = 0. __ __ __ __ __ __ __ m^3, P_{ti} = __ __ . __ mm Hg,

P_{ti} = __ __ __ . __ mm Hg, T_i = __ __ __ . __ °K, T_t = __ __ __ . __ °K

$$V_s = 0.3857\, V \left[\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}} \right] = 0. \_\_\_\_\_\_\_\_ \text{ dsm}^3 \qquad \text{Equation 6-2}$$

NONCONDENSIBLE ORGANICS CONCENTRATION

P_{ti} = __ __ mm Hg, P_t = __ __ __ mm Hg, P_{tf} = __ __ __ __ mm Hg,

T_{ti} = __ __ . __ °K, T_t = __ __ __ . __ °K , T_{tf} = __ __ __ . __ °K,

C_{tm1} = __ __ __ . __ ppm NMO, C_{tm2} = __ __ __ . __ ppm NMO,

C_{tm3} = __ __ __ . __ ppm NMO, r = __

$$C_t = \left[\frac{\frac{P_{tf}}{T_{tf}}}{\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}}} \right] \left[\frac{1}{r} \sum_{j=1}^{r} C_{tm_j} \right] = \_\_\_ . \_ \text{ ppm C} \qquad \text{Equation 6-3}$$

Figure 6.1. Calculation form for Method 25 analysis.

NONCONDESIBLE ORGANICS BLANK CONCENTRATION

$P^{t}{}_{i}$ = __ __ mm Hg, P_t = __ __ __ mm Hg, P_{tf} = __ __ __ __ mm Hg,

T_{ti} = __ __ . __ °K, T_t = __ __ __ . __ °K , T_{tf} = __ __ __ . __ °K,

C_{tmb1} = __ __ __ . __ ppm NMO, C_{tmb2} = __ __ __ . __ ppm NMO,

C_{tmb3} = __ __ __ . __ ppm NMO, r = __

$$C_{tb} = \left[\frac{\dfrac{P_{tf}}{T_{tf}}}{\dfrac{P_t}{T_t} - \dfrac{P_{ti}}{T_{ti}}} \right] \left[\frac{1}{r} \sum_{j=1}^{r} C_{tm_{bj}} \right] = \_\_\ \_\_\ \_\_ . \_\_ \text{ ppm C} \qquad \text{Equation 6-3}$$

CONDENSIBLE ORGANICS CONCENTRATION

V_v = 0. __ __ __ __ __ __ m^3, V_s = 0. __ __ __ __ __ __ dsm^3,

P_f = __ __ __ __ mm Hg, T_f = __ __ __ . __ °K,

C_{cm1} = __ __ __ __ . __ ppm CO_2, C_{cm2} = __ __ __ __ . __ ppm CO_2,

C_{cm3} = __ __ __ __ . __ ppm CO_2, q = __

$$C_c = 0.3857 \frac{V_v\ P_f}{V_s\ T_f} \left[\frac{1}{q} \sum_{k=1}^{q} C_{cm_k} \right] = \_\_\ \_\_\ \_\_\ \_\_ . \_\_ \text{ ppm C} \qquad \text{Equation 6-4}$$

Figure 6.1. Continued

CONDENSIBLE ORGANICS BLANK CONCENTRATION

V_v = 0. _ _ _ _ _ _ _ m^3, V_s = 0. _ _ _ _ _ _ _ dsm^3,

P_f = _ _ _ _ mm Hg, T_f = _ _ _ . _ °K,

C_{cmb1} = _ _ _ _ . _ ppm CO_2, C_{cmb2} = _ _ _ _ . _ ppm CO_2,

C_{cmb3} = _ _ _ _ . _ ppm CO_2, q = _

Equation 6-4

$$C_{cb} = 0.3857 \frac{V_v\ P_f}{V_s\ T_f}\left[\frac{1}{q}\sum_{k=1}^{q} C_{cm_{bk}}\right] = \_\ \_\ \_\ \_ . \_ \text{ ppm } CO_2$$

TGNMO CONCENTRATION

C_t = _ _ _ . _ ppm C, C_{tb} = _ . _ ppm C, C_c = _ _ _ _ . _ ppm C,

C_{cb} = _ _ . _ ppm C

$$C = C_t - C_{tb}^{*} + C_c - C_{cb}^{*} = \_\ \_\ \_\ \_ . \_ \text{ ppm C} \qquad \text{Equation 6-5}$$

*Note: Blank subtraction requires prior approval of the Adminstrator.

TGNMO MASS CONCENTRATION

C = _ _ _ _ . _ ppm C

$$m_c = 0.4993\ C = \_\ \_\ \_\ \_ . \_ \text{ mg C/dsm}^3 \qquad \text{Equation 6-6}$$

Figure 6.1. Continued

PERCENT RECOVERY

M = __ __ __ . __ __ g/g-mole, L = __ __ . __ ul,

P_f = __ __ __ __ . __ mm Hg, T_f = __ __ __ . __ °K,

V_v = 0. __ __ __ __ __ __ __ m^3, ρ = 0. __ __ __ __ __ kg/cc,

C_{cm} = __ __ __ __ . __ ppm C, N = __ __

$$\text{Percent Recovery} = 1.604 \frac{M}{L} \frac{V_v}{\rho} \frac{P_f}{T_f} \frac{C_{cm}}{N}$$ = __ __ __ . __ % Equation 6-7

RELATIVE STANDARD DEVIATION

$\bar{x}$ = __ __ __ . __ %, x_i = __ __ __ . __ %, n = __

$$\text{RSD} = \frac{100}{\bar{x}} \sqrt{\frac{\Sigma (x_i - \bar{x})^2}{n - 1}}$$ = __ __ . __ __ % Equation 6-8

Figure 6.1. Continued

FIELD DATA AND RESULTS TABULATION

Plant: XXXXXXXXXX

Sampling Location: XXXXXXXXXX

	Run 1	Run 2	Run 3	Blank	Audit 1	Audit 2
Date	XX/XX/XX	XX/XX/XX	XX/XX/XX	XX/XX/XX	XX/XX/XX	XX/XX/XX
Run Start Time						
Run Finish Time						
Field Data						
Sample Trap I.D.						
Sample Tank I.D.						
Sample Tank Volume, V (m^3)						
Actual Volume Sampled, V_s (dsm^{3*})						
Field Initial Barometric Pressure, P_b (cm Hg)						
Field Final Barometric Pressure (cm Hg)						
Field Initial Gauge Pressure of Tank, P_{ti} (mm Hg absolute)						
Field Final Gauge Pressure of Tank, P_t (mm Hg absolute)						
Field Initial Temperature of Tank, T_{ti} (^oK)						
Field Final Temperature of Tank, T_t (^oK)						
Laboratory Data						
Final Tank Pressure, P_{tf} (mm Hg absolute)						
Final Tank Temperature, T_{tf} (^oK)						
Noncondensible (tank) Portion - Injection #1 (area units)						
Noncondensible (tank) Portion - Injection #2 (area units)						
Noncondensible (tank) Portion - Injection #3 (area units)						
Instrument Blank (area units)						
NMO Response Factor (area units/ppm C)						

*68^{o}F -- 29.92 in. Hg (760 mm Hg)

(continued)

Figure 6.2. Recommended standard format for reporting Method 25 data and results.

	Run 1 XX/XX/XX	Run 2 XX/XX/XX	Run 3 XX/XX/XX	Blank XX/XX/XX	Audit 1 XX/XX/XX	Audit 2 XX/XX/XX
Laboratory Data (Continued)						
Volume of ICV, V_v (m^3)						
Final ICV Pressure, P_f (mm Hg absolute)						
Final ICV Temperature, T_f (^oK)						
Condensible (trap) Portion - Injection #1 (area units)						
Condensible (trap) Portion - Injection #2 (area units)						
Condensible (trap) Portion - Injection #3 (area units)						
Instrument Blank (area units)						
NMO Response Factor (area units/ppm C)						
Results						
Measured Concentration for Sample Tank, C_{tm} (ppm NMO)						
Measured Concentration for Condensate Trap, C_{cm} (ppm C)						
Noncondensible Organic Concentration (tank), C_t (ppm C)						
Condensible Organic Concentration (trap), C_c (ppm C)						
Note: Prior approval of Adminstrator required for blank substraction						
TGNMO Concentration, C (ppm C)						
Flue Gas Flow Rate (dsm^{3}h)**						
Emission Rate (mg/h)						

**From EPA Method __ testing.

Figure 6.2. Continued

Table 6.1. ACTIVITY MATRIX FOR CALCULATION CHECKS

Characteristic	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Analysis data form	All data and calculations are shown	Visually check	Complete the missing data
Calculations	Difference between check and original calculations should not exceed round-off error	Repeat all calculations starting with raw data for hand calculations; check all raw data input for computer calculations; hand calculate one sample per test	Indicate errors on calculation form, Figure 6.1

7.0 MAINTENANCE

The normal use of emission testing equipment subjects it to corrosive gases, extremes in temperature, vibration, and shock. Keeping the equipment in good operating order over an extended period of time requires knowledge of the equipment and a program of routine maintenance which is performed quarterly or after 2830 L (100 ft^3) of operation, whichever is greater. In addition to the quarterly maintenance, a yearly cleaning of pumps and metering systems is recommended. Maintenance procedures for the various components are summarized in Table 8.1 at the end of the section. The following procedures are not required, but are recommended to increase the reliability of the equipment.

7.1 Pump

Several types of pumps may be used to perform Method 25; the two most common are the fiber vane pump with in-line oiler and the diaphragm pump. The fiber vane pump requires a periodic check of the oiler jar. Its contents should be translucent; the oil should be changed if not translucent. Use the oil specified by the manufacturer. If none is specified, use SAE-10 nondetergent oil. Whenever a fiber vane pump starts to run erratically or during the yearly disassembly, the head should be removed and the fiber vanes changed. Erratic operation of a diaphragm pump is normally due to either a bad diaphragm (causing leakage) or to malfunctions of the valves, which should be cleaned annually by complete disassembly.

7.2 Rotameter

Rotameters should be disassembled and cleaned according to the manufacturer's instructions using recommended cleaning fluids every 3 months or upon erratic operation.

7.3 Manometer

The fluid in the manometers should be changed whenever there is discoloration or visible matter in the fluid, and during the yearly disassembly.

7.4 Sampling Train

All remaining sampling train components should be visually checked every 3 months and completely disassembled and cleaned or replaced yearly. Many items, such as quick disconnects, should be replaced whenever damaged rather than checked periodically. Normally, the best procedure for maintenance in the field is to have on hand another entire sampling system, including a pump, probe, U-tubes, filter holders, sample tanks, and heated sample line rather than replacing individual components. It is recommended that the U-tubes be filled with nitrogen after burnout to reduce oxidation of the metal surface. The sample tanks should be clean and dry when being stored between tests.

7.5 Gas Chromatograph and NDIR

Maintenance activities and schedules for gas chromatographs and NDIRs are make and model specific. It is recommended that the analyst consult the operator's manual for instructions relative to maintenance practices and procedures.

Table 7.1. ACTIVITY MATRIX FOR EQUIPMENT MAINTENANCE CHECKS

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Fiber vane pump	In-line oiler free of leaks	Periodically check oiler jar; remove head and change fiber vanes	Replace as needed
Diaphragm pump	Leak-free valves functioning properly	Clean valves during yearly disassembly	Replace when leaking or malfunctioning
Rotameter	Clean and no erratic behavior	Clean every 3 mo. or whenever ball does not move freely	Replace
Manometer	No discoloration or visible matter in the fluid	Check periodically and during disassembly	Replace parts as needed
Sampling train components	No damage	Visually check every 3 mo.; completely disassemble and clean or replace yearly	If failure noted, replace appropriate components
Gas chromatograph and NDIR	See owner's manual	See owner's manual	See owner's manual

8.0 AUDITING PROCEDURES

An audit is an independent assessment of data quality. Independence is achieved if the individual(s) performing the audit and their standards and equipment are different from the regular field team and their standards and equipment. Routine quality assurance checks by a field team are necessary to generate good quality data, but they are not part of the auditing procedure. Table 8.1 at the end of this section summarizes the quality assurance functions for auditing.

Based on the requirements of Method 25 and the results of collaborative testing of other EPA Test Methods, one performance audit is required when testing for compliance for Standards of New Source Performance (and as required by other government agencies) and is recommended when testing for other purposes; and a second performance audit is recommended. The two performance audits are:

1. An audit of the sampling and analysis of Method 25 is required for NSPS and recommended for other purposes.
2. An audit of the data processing is recommended.

It is suggested that a systems audit be conducted as specified by the quality assurance coordinator in addition to these performance audits. The two performance audits and the systems audit are described in detail in Subsections 8.1 and 8.2, respectively.

8.1 Performance Audits

Performance audits are conducted to evaluate quantitatively the quality of data produced by the total measurement system (sample collection, sample analysis, and data processing). It is required that a cylinder gas performance audit be performed once during every NSPS test utilizing Method 25 and it is recommended that a cylinder gas audit be performed once during any enforcement source test utilizing Method 25 conducted under regulations other than NSPS.

8.1.1 *Performance Audit of the Field Test* - As stated in Section 4.5 of Method 25 (40 CFR 60, Appendix A) and the "Instructions for the Sampling and Analysis of Total Gaseous Nonmethane Organics from Quality Assurance Audit Cylinders using EPA Method 25 Procedures" (supplied with the EPA audit gas cylinders), a set of two audit samples are to be collected in the field (not laboratory) from two different concentration gas cylinders at the same time the compliance test samples are being collected. The two audit samples are then analyzed concurrently and in exactly the same manner as the compliance samples to evaluate the tester's and analyst's technique and the instrument calibration. The information required to document the collection and analysis of the audit samples has been included on the example data sheets shown later in Figures 8.1 and 8.3. The audit analyses shall agree within 20 percent of the actual cylinder concentrations.

The operator of the affected facility is responsible for informing local, state, and federal agencies of the test program and of the test schedule. Therefore, the operator of the affected facility is responsible for requesting (and handling of) the audit samples from the agency responsible for observing the compliance test. The tester is responsible for informing his client of the details of any method used and any audit samples required to validate the compliance test. These details are usually part of a pretest meeting, but many times pretest meetings are not necessary depending on the scope of the work and permit requirements.

The facility (or tester) may obtain audit cylinders by contacting the agency responsible for observing and/or evaluating the compliance test and informing the agency the time and location of the compliance test. This should be done at least 14 days prior to the test date. The responsible agency will contact: U.S. Environmental Protection Agency, Atmospheric Research and Exposure Laboratory, Quality Assurance Division (MD-77), Research Triangle Park, North Carolina 27711 and have the cylinders shipped to the specified site.

Responsibilities of the Audit Supervisor - The primary responsibilities of the audit supervisor are to ensure that the proper audit gas cylinders are ordered and safe-guarded, and to interpret the results obtained by the analyst.

When notified by the testing company that a test is to be conducted, the audit supervisor will order the proper cylinders from the EPA's Quality Assurance Division. Generally the audit cylinders will be shipped (at EPA's expense) directly to the specified site. However, if the audit supervisor will be on-site during the compliance test, the audit cylinders may be shipped to the testing company for transport to the sampling site. Since the audit cylinders are sealed by EPA, the testing firm will not be allowed to collect any audit gas without breaking the seal. The audit gas concentration(s) should be in the range of 50% below to 100% above the applicable standard. If two cylinders are not available, then one cylinder can be used.

The audit supervisor must ensure that the audit gas cylinder(s) are shipped to the correct address, and to prevent vandalism, verify that they are stored in a safe location both before and after the audit. Also, the audit cylinders should not be analyzed when the pressure drops below 200 psi. The audit supervisor then ensures that the audits are conducted as described below. At the conclusion of the collection of the audit samples, the cylinders are then returned to shipping laboratory as per the instructions supplied with the cylinders at the expense of the facility. If the tester is to transport the audit cylinders to his home laboratory for shipment back to the EPA/QAD contractor, the audit supervisor will seal both cylinders to ensure that additional audit sample gas cannot be collected without breaking the seal.

The audit supervisor must also interpret the audit results. Indication of acceptable results may be obtained immediately by reporting the audit compliance test results in ppm by telephone to the responsible enforcement agency. The tester must also include the results of both audit samples, their identification numbers, and the analyst's name along with the results of the compliance determination samples in the appropriate reports to the EPA regional office or the appropriate enforcement agency during the 30-day period.

When the measured concentration agrees within 20% of the true value, the audit results are considered acceptable. Failure to meet the 20-percent specification may require reanalysis of the audit samples and compliance test samples, reauditing, or retests until the audit problems are resolved. However, if the audit results do not affect the compliance or noncompliance status of the affected facility, the Administrator (enforcement agency) may waive the reanalysis, further audits, or retest requirements and accept the results of the compliance test. For example, if the audit results average 38.6% low, the compliance results would be divided by (1 - 0.386) to determine the correlated effect. If the audit results average 58.3% high, the compliance sample results would be divided by (1 + 0.583) to determine the effect. When the compliance status of the source is the same with and without the correlated value, then the responsible agency may accept the results of the test. While steps are being taken to resolve audit analysis problems, the Administrator may also choose to use the data to determine the compliance or noncompliance of the affected facility.

The same analysts, analytical reagents, and analytical system shall be used for the compliance samples and the EPA audit samples; if this condition is met, and the same testing firm is collecting other sets of compliance samples, auditing of subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both enforcement agencies.

During the audit, the audit supervisor should record the coded cylinder number(s) and cylinder pressure(s) on the "Audit Report" form, Figure 8.1. The individual being audited must not be told the actual audit concentrations or the calculated audit percent accuracy.

On-site Collection of Audit Sample(s) - The cylinder gas performance audit must be conducted in the field (not laboratory) at the same time the compliance test samples are being taken. A maximum of 5L of audit gas is to be used for each test run unless multiple tanks are required for sampling. The tester will be required to supply a two-stage regulator (CGA - 350), a glass manifold or Teflon tee connection and other suitable Swagelok fittings (they are not supplied) for use with the audit gas cylinder. The recommended procedures for conducting the on-site audit sample collection are as follows:

1. The audit supervisor (agency representative) should verify that the seal affixed by shipping or supplying laboratory is still intact. After the seal has been checked by the audit supervisor, the tester may break the seal. However, if the audit supervisor is not present at the time of the audit, the tester may break the seal and proceed with the audit.
2. The tester should set up the Method 25 sampling train and perform the leak check.
3. The audit gas from the cylinder has to be sampled at atmospheric pressure either from a glass manifold or through a Teflon tee connection. This can be done by attaching both the cylinder and the probe of the Method 25 sampling train to two of the manifold or tee connections while excess gas flows out through the remaining connection as shown in Figure 8.2. This can be accomplished by starting the cylinder gas flow into the manifold or tee with the sampling train flow turned off. Then, turn on the sampling train flow while adjusting the flow from the audit cylinder to ensure excess audit gas flow from the manifold or tee. After the proper sampling flow rate has been obtained in the sampling train, adjust the audit cylinder so only a few cubic centimeters of excess gas is discharged from the manifold or tee. The tester must ensure that the audit gas is conserved.
4. Use the same sampling flow rate and sample volume as used for field test samples. When a constant flow rate can no longer be maintained by the sampling train, it should be turned off and then the audit cylinder shut off. Ensure that the audit cylinder is closed tight to prevent leakage. If the compliance test requires more than one sample tank to complete a run, the audit should use the same number of tanks required by the average run.
5. The same procedures are repeated for the second audit cylinder using a separate sampling train.
6. The sampling trains containing the audit samples should be stored and shipped in the same manner as and along with the field test samples.
7. In all cases, it is recommended that the audit supervisor reseal the audit cylinders to ensure no tampering. However, if the test firm is to return the cylinder to shipping or supplying laboratory, it is mandatory that the

audit cylinders are resealed by the audit supervisor.

8. The audit cylinders are to be returned immediately after the test to the EPA/QAD contractor at the cost of the facility (or tester if applicable) either by ground transportation or air cargo. They are not to be shipped collect.

Analysis of Audit Sample(s) - Analyze the collected audit sample fractions (condensibles and noncondensibles) at the same time as the Method 25 compliance test samples. Follow the procedures described in the method for sample analysis, calibration, and calculations. The same analysts, analytical reagents, and analytical system shall be used for both the compliance test samples and the EPA audit samples.

Reporting of Audit Sample(s) Results - The reporting of the audit results should be the responsibility of the tester to ensure taht the data is acceptable and valid. The audit sample results are to be reported to the responsible agency by the testing firm in terms of condensibles (U-trap fraction), noncondensibles (tank fraction), and total (sum of both fractions) as parts-per-million carbon (ppm C). The agency will in turn report the results to the EPA/QAD contractor for continuing evaluation of the Method 25 audit program. Additionally, the tester must supply document in the test report, the results of both audit samples as described above, their identification numbers, and the analyst's name along with the results of the compliance determination samples. The operator of the affected facility is responsible for the dissemination of any compliance sample results and the correct distribution of reports to the EPA regional office or the appropriate enforcement agency during the 30-day period for which the audit samples represent.

8.1.2 *Performance Audit of Data Processing* - Calculation errors are prevalent in processing data. Data processing errors can be determined by auditing the recorded data on the field and laboratory forms. The original and audit (check) calculations should agree within round-off error; if not, all of the remaining data should be checked. The data processing may also be audited by providing the testing laboratory with specific data sets (exactly as would appear in the field), and by requesting that the data calculation be completed and that the results be returned to the agency. This audit is useful in checking both computer programs and manual methods of data processing.

8.2 Systems Audit

A systems audit is an on-site, qualitative inspection and review of the total measurement system (sample collection, sample analysis, etc.). Initially, a systems audit is recommended for each enforcement source test, defined here as a series of three runs at one source. After the test team gains experience with the method, the frequency of auditing may be reduced -- for example, to once every four tests.

The auditor should have extensive background experience in source sampling, specifically with the measurement system being audited. The functions of the auditor are summarized below:

1. Inform the testing team of the results from previous audits, specifying any area(s) that need special attention or improvement.
2. Observe procedures and techniques of the field team during sample collection.
3. Check/verify records of apparatus calibration checks and quality control used in the laboratory analysis of control samples from previous source

tests, where applicable.

4. Record the results of the audit, and forward them with comments to the test team management so that appropriate corrective action may be initiated.

While on site, the auditor observes the source test team's overall performance, including the following specific operations:

1. Setting up and leak testing the sampling train.
2. Collecting the sample at a constant rate at the specified flow rate.
3. Conducting the final leak check and recovery of the samples.
4. Sample documentation procedures, sample recovery, and preparation of samples for shipment.

Figure 8.3 is a suggested checklist for the auditor.

AUDIT REPORT

Part A. - To be filled out by organization supplying audit cylinders.

1. Organization supplying audit sample(s) and shipping address
____
2. Audit supervisor, organization, and phone number
____
3. Shipping instructions: Name, Address, Attention
____
4. Guaranteed arrival date for cylinders - __________
5. Planned shipping date for cylinders - __________
6. Details on audit cylinders from last analysis

	Low conc.	High conc.
a. Date of last analysis......	....	....
b. Cylinder number............	....	....
c. Cylinder pressure, psi.....	....	....
d. Audit gas(es)/balance gas..	N_2	N_2
e. Audit gas(es), ppm.........	....	....
f. Cylinder construction......	Aluminum	Aluminum

Part B. - To be filled out by audit supervisor.

1. Process sampled __________
2. Audit location __________
3. Name of individual audit __________
4. Audit date __________
5. Audit cylinders sealed __________
6. Audit results:

	Low conc. cylinder	High conc. cylinder
a. Cylinder number...........................	....	....
b. Cylinder pressure before audit, psi.........	....	....
c. Cylinder pressure after audit, psi..........	....	....
d. Measured concentration, ppm C		
U-tube fraction	....	....
Tank fraction	....	....
Total concentration	....	....
e. Actual audit concentration, ppm C		
f. Audit accuracy:[1]		
Low conc. cylinder..........................	....	....
High conc. cylinder.........................	....	....
Percent accuracy[1] = $\frac{\text{Measured Conc. - Actual Conc.}}{\text{Actual Conc.}} \times 100$		
g. Problems detected (if any).................	....	....

[1] The audit accuracy is calculated on the total concentration only.

Figure 8.1. Field audit report form.

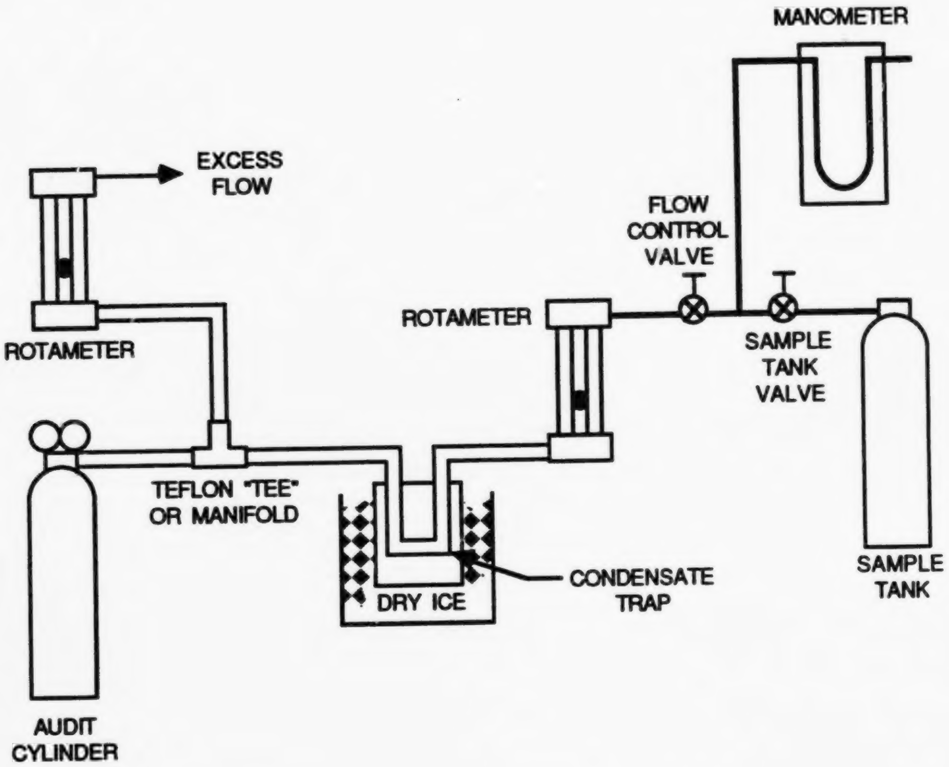
MANOMETER
EXCESS FLOW
FLOW CONTROL VALVE
ROTAMETER
ROTAMETER
SAMPLE TANK VALVE
TEFLON "TEE" OR MANIFOLD
DRY ICE
CONDENSATE TRAP
SAMPLE TANK
AUDIT CYLINDER

Figure 8.2. Schematic of Method 25 audit system.

Yes	No	Comments	Operation
			PRESAMPLING PREPARATION
			1. Knowledge of process operations
			2. Calibration of pertinent equipment, in particular, temperature readouts and flowmeters
			3. Selection and checkout of equipment for proper sampling techniques
			ON-SITE MEASUREMENTS
			4. Sampling system properly assembled
			5. Sampling system leak check acceptable
			6. Sample probe and filter at proper temperature
			7. Sample system purged properly
			8. Constant rate sampling properly conducted
			9. Heater systems maintained at proper temperatures
			10. Proper number of samples & sampling time
			11. Recording of pertinent process conditions during sample collection, samples properly identified, and calculations properly conducted
			POSTSAMPLING
			12. Results of audit (± 20% or other value)
			13. Oxidation catalyst efficiency test acceptable
			14. Reduction catalyst efficiency test acceptable
			15. NMO linearity and calibration test acceptable
			16. CO$_2$ linearity and calibration test acceptable
			17. NMO analyzer performance test acceptable
			18. Condensible organic recovery system leak check
			19. System background test acceptable
			20. Temperature, volumes, pressures, and concentrations properly recorded
			21. Analytical results properly calculated

COMMENTS

Figure 8.3. Method 25 checklist to be used by auditors.

Table 8.1. ACTIVITY MATRIX FOR AUDITING PROCEDURES

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Performance audit of analytical phase	Measured relative error of audit samples less than 20% for both samples	Frequency: Once during every enforcement source test* Method: Measure audit samples and compare results to true values	Review operating technique and repeat audit, repeat test, reject test, or accept results
Data processing errors	Original and checked calculations agree within round-off error	Frequency: Once during every enforcement source test* Method: Independent calculations starting with recorded data	Check and correct all data from the audit period represented by the checked data
Systems audit--observance of technique	Operational technique as described in this section of the Handbook	Frequency: Once during every enforcement source test* until experience gained, then every fourth test Method: Observation of techniques assisted by audit checklist, Figure 8.3	Explain to test team their deviations from reccommended techniques and note on Fig. 8.3

*As defined here, a source test for enforcement of the NSPS comprises a series of runs at one source. Source tests for purposes other than enforcement of NSPS may be audited at the frequency determined by the applicable group.

9.0 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

To achieve data of desired quality, two essential considerations are necessary: (1) the measurement process must be in a state of statistical control at the time of the measurement, and (2) the systematic errors, when combined with the random variation (errors or measurment), must result in an acceptable uncertainty. As evidence in support of good quality data, it is necessary to perform quality control checks and independent audits of the measurement process; to document these data; and to use materials, instruments, and measurement procedures that can be traced to an apropriate standard of reference.

Data must be routinely obtained by repeat measurements of standard reference samples (primary, secondary, and/or working standards) and the establishment of a condition of process control. The working calibration standards should be traceable to standards of higher accuracy. It is recommended, but not required, that the analyst use a NBS-SRM for propane to make the analysis traceable to an NBS Standard Reference Material.

Audit samples (as discussed in Section 3.17.8) must be used to validate test results for compliance determination purposes and are recommendeed as an independent check on the measurement process when the method is performed for other purposes. This makes all the compliance determination samples traceable to the same audit source at EPA.

10.0 REFERENCE METHOD

Method 25 - Determination of Total Gaseous NonMethane Organic Emissions as Carbon

1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of volatile organic compounds (VOC) as total gaseous nonmethane organics (TGNMO) as carbon in source emissions. Organic particulate matter will interfere with the analysis, and, therefore, a particulate filter is required. The minimum detectable concentration for the method is 50 ppm as carbon.

When carbon dioxide (CO_2) and water vapor are present together in the stack, they can produce a positive bias in the sample. The magnitude of the bias depends on the concentration of CO_2 and water vapor. As a guideline, multiply the CO_2 concentration, expressed as volume percent, times the water vapor concentration. If this product does not exceed 100, the bias can be considered insignificant. For example, the bias is not significant for a source having 10 percent CO_2 and 10 percent water vapor, but it would be significant for a source near the detection limit having 10 percent CO_2 and 20 percent water vapor.

This method is not the only method that applies to the measurement of TGNMO. Costs, logistics, and other practicalities of source testing may make other test methods more desirable for measuring VOC contents of certain effluent streams. Proper judgment is required in determining the most applicable VOC test method. For example, depending upon the molecular weight of the organics in the effluent stream, a totally automated semicontinuous nonmethane organics (NMO) analyzer interfaced directly to the source may yield accurate results. This approach has the advantage of providing emission data semicontinuously over an extended time period.

Direct measurement of an effluent with a flame ionization detector (FID) analyzer may be appropriate with prior characterization of the gas stream and knowledge that the detector responds predictably to the organic compounds in the stream. If present, methane (CH_4) will, of course, also be measured. The FID can be applied to the determination of the mass concentration of the total molecular structure of the organic emissions under any of the following limited conditions: (1) where only one compound is known to exist; (2) when the organic compounds consist of only hydrogen and carbon; (3) where the relative percentages of the compounds are known or can be determined, and the FID responses to the compounds are known; (4) where a consistent mixture of the compounds exists before and after emission control and only the relative concentrations are to be assessed; or (5) where the FID can be calibrated against mass standards of the compounds emitted (solvent emissions, for example).

Another example of the use of a direct FID is as a screening method. If there is enough information available to provide a rough estimate of the analyzer accuracy, the FID analyzer can be used to determine the VOC content of an uncharacterized gas stream. With a sufficient buffer to account for possible inaccuracies, the direct FID can be a useful tool to obtain the desired results without costly exact determination. In situations where a qualitative/ quantitative analysis of an effluent stream is desired or required, a gas chromatographic FID system may apply. However, for sources emitting numerous organics, the time and expense of this approach will be formidable.

1.2 Principle. An emission sample is withdrawn from the stack at a constant rate through a heated filter and a chilled condensate trap by means of an evacuated sample tank. After sampling is completed, the TGNMO are determined by independently analyzing the condensate trap and sample tank fractions and combining the analytical results. The organic content of the condensate trap fraction is determined by oxidizing the NMO to CO_2 and quantitatively collecting the effluent in an evacuated vessel; then a portion of the CO_2 is reduced to CH_4 and measured by an FID. The organic content of the sample tank fraction is measured by injecting a portion of the sample into a gas chromatographic column to separate the NMO from carbon monoxide (CO). CO_2. and CH_4; the NMO are oxidized to CO_2. reduced to CH_4. and measured by an FID. In this manner, the variable response of the FID associated with different types of organics is eliminated.

2. Apparatus

2.1 Sampling. The sampling system consists of a heated probe, heated filter, condensate trap, flow control system, and sample tank (Figure 25-1). The TGNMO sampling equipment can be constructed from commercially available components and components fabricated in a machine shop. The following equipment is required:

2.1.1 Heated Probe. 6.4-mm (1/4-in.) OD stainless steel tubing with a heating system capable of maintaining a gas temperature at the exit end of at least 129°C (265°F). The probe shall be equipped with a thermocouple at the exit end to monitor the gas temperature.

A suitable probe is shown in Figure 25-1. The nozzle is an elbow fitting attached to the front end of the probe while the thermocouple is inserted in the side arm of a tee fitting attached to the rear of the probe. The probe is wrapped with a suitable length of high temperature heating tape, and then covered with two layers of glass cloth insulation and one layer of aluminum foil.

NOTE: If it is not possible to use a heating system for safety reasons, an unheated system with an in-stack filter is a suitable alternative.

2.1.2 Filter Holder. 25-mm (15/16-in.) ID Gelman filter holder with stainless steel body and stainless steel support screen with the Viton O-ring replaced by a Teflon O-ring.

NOTE: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1.3 Filter Heating System. A metal box consisting of an inner and an outer shell separated by insulating material with a heating element in the inner shell capable of maintaining a gas temperature at the filter of 121 ± 3°C (250 ± 5°F).

A suitable heating box is shown in Figure 25-2. The outer shell is a metal box that measures 102 mm x 280 mm x 292 mm (4 in. x 11 in. x 11 1/2 in.). while the inner shell is a metal box measuring 76 mm x 229 mm x 241 mm (3 in. x 9 in. x 9 1/2 in.). The inner box is supported by 13-mm (1/2-in.) phenolic rods. The void space between the boxes is filled with fiberfrax insulation which is sealed in place by means of a silicon rubber bead around the upper sides of the box. A removable lid made in a similar manner, with a 25-mm (1-in.) gap between the parts, is used to cover the heating chamber.

The inner box is heated with a 250-watt cartridge heater, shielded by a stainless steel shroud. The heater is regulated by a thermostatic temperature controller which is set to maintain a temperature of 121°C as measured by a thermocouple in the gas line just before the filter. An additional thermocouple is used to monitor the temperature of the gas behind the filter.

2.1.4 Condensate Trap. 9.5-mm (3/8-in.) OD 316 stainless steel tubing bent into a U-shape. Exact dimensions are shown in Figure 25-3. The tubing shall be packed with coarse quartz wool, to a density of approximately 0.11 g/cc before bending. While the condensate trap is packed with dry ice in the Dewar, an ice bridge may form between the arms of the condensate trap making it difficult to remove the condensate trap. This problem can be prevented by attaching a steel plate between the arms of the condensate trap in the same plane as the arms to completely fill the intervening space.

2.1.5 Valve. Stainless steel shut-off valve for starting and stopping sample flow.

2.1.6 Metering Valve. Stainless steel control valve for regulating the sample flow rate through the sample train.

2.1.7 Rotameter. Glass tube with stainless steel fittings, capable of measuring sample flow in the range of 60 to 100 cc/min.

2.1.8 Sample Tank. Stainless steel or aluminum tank with a minimum volume of 4 liters.

2.1.9 Mercury Manometer or Absolute Pressure Gauge. Capable of measuring pressure to within 1 mm Hg in the range of 0 to 900 mm.

2.1.10 Vacuum Pump. Capable of evacuating to an absolute pressure of 10 mm Hg.

2.2. Condensate Recovery Apparatus. The system for the recovery of the organics captured in the condensate trap consists of a heat source, oxidation catalyst, nondispersive infrared (NDIR) analyzer and an intermediate collection vessel (ICV). Figure 25-4 is a schematic of a typical system. The system shall be capable of proper oxidation and recovery, as specified in Section 5.1. The following major components are required:

2.2.1 Heat Source. Sufficient to heat the condensate trap (including connecting tubing) to a temperature of 200°C. A system using both a heat gun and an electric tube furnace is recommended.

2.2.2 Heat Tape. Sufficient to heat the connecting tubing between the water trap and the oxidation catalyst to 100°C.

2.2.3. Oxidation Catalyst. A suitable length of 9.5-mm (3/8-in.) OD Inconel 600 tubing packed with 15 cm (6 in.) of 3.2-mm (1/8-in.) diameter 19 percent chromia on alumina pellets. The catalyst material is packed in the center of the catalyst tube with quartz wool packed on either end to hold it in place. The catalyst tube shall be mounted vertically in a 650°C tube furnace.

2.2.4 Water Trap. Leak proof, capable of removing moisture from the gas stream.

2.2.5 Syringe Port. A 6.4-mm (1/4-in.) OD stainless steel tee fitting with a rubber septum placed in the side arm.

2.2.6 NDIR Detector. Capable of indicating CO_2 concentration in the range of zero to 5 percent, to monitor the progress of combustion of the organic compounds from the condensate trap.

2.2.7 Flow-Control Valve. Stainless steel, to maintain the trap conditioning system near atmospheric pressure.

2.2.8 Intermediate Collection Vessel. Stainless steel or aluminum, equipped with a female quick connect. Tanks with nominal volumes of at least 6 liters are recommended.

2.2.9 Mercury Manometer or Absolute Pressure Gauge. Capable of measuring pressure to within 1 mm Hg in the range of 0 to 900 mm.

2.2.10 Syringe. 10-ml gas-tight, glass syringe equipped with an appropriate needle.

2.3 NMO Analyzer. The NMO analyzer is a gas chromatograph (GC) with backflush capability for NMO analysis and is equipped with an oxidation catalyst, reduction catalyst, and FID. Figures 25-5 and 25-6 are schematics of a typical NMO analyzer. This semicontinuous GC/FID analyzer shall be capable of: (1) separating CO, CO_2, and CH_4 from NMO, (2) reducing the CO_2 to CH_4 and quantifying as CH_4. and (3) oxidizing the NMO to CO_2, reducing the CO_2 to CH_4 and quantifying as CH_4. according to Section 5.2. The analyzer consists of the following major components:

2.3.1 Oxidation Catalyst. A suitable length of 9.5-mm (3/8-in.) OD Inconel 600 tubing packed with 5.1 cm (2 in.) of 19 percent chromia on 3.2-mm (1/8-in.) alumina pellets. The catalyst material is packed in the center of the tube supported on either side by quartz wool. The catalyst tube must be mounted vertically in a 650°C furnace.

2.3.2 Reduction Catalyst. A 7.6-cm (3-in.) length of 6.4-mm (1/4-in.) OD Inconel tubing fully packed with 100-mesh pure nickel powder. The catalyst tube must be mounted vertically in a 400°C furnace.

2.3.3 Separation Column(s). A 30-cm (1-ft) length of 3.2-mm (1/8-in.) OD stainless steel tubing packed with 60/80 mesh Unibeads 1S followed by a 61-cm (2-ft) length of 3.2-mm (1/8-in.) OD stainless steel tubing packed with 60/80 mesh Carbosieve G. The Carbosieve and Unibeads columns must be baked separately at 200°C with carrier gas flowing through them for 24 hours before initial use.

2.3.4 Sample Injection System. A 10-port GC sample injection valve fitted with a sample loop properly sized to interface with the NMO analyzer (1-cc loop recommended).

2.3.5 FID. An FID meeting the following specifications is required:

2.3.5.1 Linearity. A linear response (± 5 percent) over the operating range as demonstrated by the procedures established in Section 5.2.3.

2.3.5.2 Range. A full scale range of 10 to 50,000 ppm CH_4. Signal attenuators shall be available to produce a minimum signal response of 10 percent of full scale.

2.3.6 Data Recording System. Analog strip chart recorder or digital integration system compatible with the FID for permanently recording the analytical results.

2.4 Other Analysis Apparatus.

2.4.1 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 1 mm Hg.

2.4.2 Thermometer. Capable of measuring the laboratory temperature to within 1°C.

2.4.3 Vacuum Pump. Capable of evacuating to an absolute pressure of 10 mm Hg.

2.4.4 Syringes. 10-ul and 50-ul liquid injection syringes.

2.4.5 Liquid Sample Injection Unit. 316 SS U-tube fitted with an injection septum, see Figure 25-7.

3. Reagents

3.1 Sampling. The following are required for sampling:

3.1.1 Crushed Dry Ice.

3.1.2 Coarse Quartz Wool. 8 to 15 um.

3.1.3 Filters. Glass fiber filters, without organic binder.

3.2 NMO Analysis. The following gases are needed:

3.2.1 Carrier Gases. Zero grade helium (He) and oxygen (O_2) containing less than 1 ppm CO_2 and less than 0.1 ppm C as hydrocarbon.

3.2.2 Fuel Gas. Zero grade hydrogen (H_2), 99.999 percent pure.

3.2.3 Combustion Gas. Zero grade air or O_2 as required by the detector.

3.3 Condensate Analysis. The following gases are needed:

3.3.1 Carrier Gas. Zero grade air, containing less than 1 ppm C.

3.3.2 Auxiliary O_2. Zero grade O_2, containing less than 1 ppm C.

3.3.3 Hexane. ACS grade, for liquid injection.

3.3.4 Decane. ACS grade, for liquid injection.

3.4 Calibration. For all calibration gases, the manufacturer must recommend a maximum shelf life for each cylinder (i.e., the length of time the gas concentration is not expected to change more than ± 5 percent from its certified value). The date of gas cylinder preparation, certified organic concentration, and recommended maximum shelf life must be affixed to each cylinder before shipment from the gas manufacturer to the buyer. The following calibration gases are required:

3.4.1 Oxidation Catalyst Efficiency Check Calibration Gas. Gas mixture standard with nominal concentration of 1 percent methane in air.

3.4.2 FID Linearity and NMO Calibration Gases. Three gas mixture standards with nominal propane concentrations of 20 ppm, 200 ppm, and 3000 ppm, in air.

3.4.3 CO_2 Calibration Gases. Three gas mixture standards with nominal CO_2 concentrations of 50 ppm, 500 ppm, and 1 percent, in air.
NOTE: Total NMO of less than 1 ppm required for 1 percent mixture.

3.4.4 NMO Analyzer System Check Calibration Gases. Four calibration gases are needed as follows:

3.4.4.1 Propane Mixture. Gas mixture standard containing (nominal) 50 ppm CO, 50 ppm CH_4, 2 percent CO_2, and 20 ppm C_3H_8, prepared in air.

3.4.4.2 Hexane. Gas mixture standard containing (nominal) 50 ppm hexane in air.

3.4.4.3 Toluene. Gas mixture standard containing (nominal) 20 ppm toluene in air.

3.4.4.4 Methanol. Gas mixture standard containing (nominal) 100 ppm methanol in air.

4. Procedure

4.1 Sampling.

4.1.1 Cleaning Sampling Equipment. Before its initial use and after each subsequent use, a condensate trap should be thoroughly cleaned and checked to insure that it is not contaminated. Both cleaning and checking can be accomplished by installing the trap in the condensate recovery system and treating it as if it were a sample. The trap should be heated as described in the final paragraph of Section 4.3.3. A trap may be considered clean when the CO_2 concentration in its effluent gas drops below 10 ppm. This check is optional for traps that have been used to collect samples which were then recovered according to the procedure in Section 4.3.3.

4.1.2 Sample Tank Evacuation and Leak Check. Evacuate the sample tank to 10 mm Hg absolute pressure or less. Then close the sample tank valve, and allow the tank to sit for 60 minutes. The tank is acceptable if no change in tank vacuum is noted. The evacuation and leak check may be conducted either in the laboratory or the field. The results of the leak check should be included in the test report.

4.1.3 Sample Train Assembly. Just before assembly, measure the tank vacuum using a mercury U-tube manometer or absolute pressure gauge. Record this vacuum, the ambient temperature, and the barometric pressure at this time. Close the sample tank valve and assemble the sampling system as shown in Figure 25-1. Immerse the condensate trap body in dry ice. The point where the inlet tube joins the trap body should be 2.5 to 5 cm above the top of the dry ice.

4.1.4 Pretest Leak Check. A pretest leak check is required. Calculate or measure the approximate volume of the sampling train from the probe trip to the sample tank valve. After assembling the sampling train, plug the probe tip, and make certain that the sample tank valve is closed. Turn on the vacuum pump, and evacuate the sampling system from the probe tip to the sample tank valve to an absolute pressure of 10 ppm Hg or less. Close the purge valve, turn off the pump, wait a minimum period of 5 minutes, and recheck the indicated vacuum. Calculate the maximum allowable pressure change based on a leak rate of 1 percent of the sampling rate using Equation 25-1, Section 6.2. If the measured pressure change exceeds the calculated limit, correct the problem before beginning sampling. The results of the leak check should be included in the test report.

4.1.5 Sample Train Operation. Unplug the probe tip, and place the probe into the stack such that the probe is perpendicular to the duct or stack axis; locate the probe tip at a single preselected point of average velocity facing away from the direction of gas flow. For stacks having a negative static pressure, seal the sample port sufficiently to prevent air in-leakage around the probe. Set the probe temperature controller to 129°C (265°F) and the filter temperature controller to 121°C (250°F). Allow the probe and filter to heat for about 30 minutes before purging the sample train.

Close the sample valve, open the purge valve, and start the vacuum pump. Set the flow rate between 60 and 100 cc/min, and purge the train with stack gas for at least 10 minutes. When the temperatures at the exit ends of the probe and filter are within their specified range, sampling may begin.

Check the dry ice level around the condensate trap, and add dry ice if necessary. Record the clock time. To begin sampling, close the purge valve and stop the pump. Open the sample valve and the sample tank valve. Using the flow control valve, set the flow through the sample train to the proper rate. Adjust the flow rate as necessary to maintain a constant rate (±10 percent) throughout the duration of the sampling period. Record the sample tank vacuum and flowmeter setting at 5-minute intervals. (See Figure 25-8.) Select a total sample time greater than or equal to the minimum sampling time specified in the applicable subpart of the regulation; end the sampling when this time period is reached or when a constant flow rate can no longer be maintained because of reduced sample tank vacuum.

NOTE: If sampling had to be stopped before obtaining the minimum sampling time (specified in the applicable subpart) because a constant flow rate could not be maintained, proceed as follows: After closing the sample tank valve, remove the used sample tank from the sampling train (without disconnecting other portions of the sampling train). Take another evacuated and leak-checked sample tank, measure and record the tank vacuum, and attach the new tank to the sampling train. After the new tank is attached to the sample train, proceed with the sampling until the required minimum sampling time has been exceeded.

4.2 Sample Recovery. After sampling is completed, close the flow control valve, and record the final tank vacuum; then record the tank temperature and barometric pressure. Close the sample tank valve, and disconnect the sample tank from the sample system. Disconnect the condensate trap at the flowmetering system, and tightly seal both ends of the condensate trap. Do not include the probe from the stack to the filter as part of the condensate sample. Keep the trap packed in dry ice until the samples are returned to the laboratory for analysis. Ensure that the test run number is properly identified on the condensate trap and the sample tank(s).

4.3 Condensate Recovery. See Figure 25-9. Set the carrier gas flow rate, and heat the catalyst to its operating temperature to condition the apparatus.

4.3.1 Daily Performance Checks. Each day before analyzing any samples, perform the following tests:

4.3.1.1 Leak Check. With the carrier gas inlets and the flow control valve closed, install a clean condensate trap in the system, and evacuate the system to 10 mm Hg absolute pressure or less. Close the vacuum pump valve and turn off the vacuum pump. Monitor the system pressure for 10 minutes. The system is acceptable if the pressure change is less than 2 mm Hg.

4.3.1.2 System Background Test. Adjust the carrier gas and auxiliary oxygen flow rate to their normal values of 100 cc/min and 150 cc/min, respectively, with the sample recovery valve in vent position. Using a 10-ml syringe withdraw a sample from the system effluent through the syringe port. Inject this sample into the NMO analyzer, and measure the CO_2 content. The system background is acceptable if the CO_2 concentration is less than 10 ppm.

4.3.1.3 Oxidation Catalyst Efficiency Check. Conduct a catalyst efficiency test as specified in Section 5.1.2 of this method. If the criterion of this test cannot be met, make the necessary repairs to the system before proceeding.

4.3.2 Condensate Trap CO_2 Purge and Sample Tank Pressurization. After sampling is completed, the condensate trap will contain condensed water and organics and a small volume of sampled gas. This gas from the stack may contain a significant amount of CO_2 which must be removed from the condensate trap before the sample is recovered. This is accomplished by purging the condensate trap with zero air and collecting the purged gas in the original sample tank.

Begin with the sample tank and condensate trap from the test run to be analyzed. Set the four-port valve of the condensate recovery system in the CO_2 purge position as shown in Figure 25-9. With the sample tank valve closed, attach the sample tank to the sample recovery system. With the sample recovery valve in the vent position and the flow control valve fully open, evacuate the manometer or pressure gauge to the vacuum of the sample tank. Next, close the vacuum pump valve, open the sample tank valve, and record the tank pressure.

Attach the dry-ice-cooled condensate trap to the recovery system, and initiate the purge by switching the sample recovery valve from vent to collect position. Adjust the flow control valve to maintain atmospheric pressure in the recovery system. Continue the purge until the CO_2 concentration of the trap effluent is less than 5 ppm. CO_2 concentration in the trap effluent should be measured by extracting syringe samples from the recovery system and analyzing the samples with the NMO analyzer. This procedure should be used only after the NDIR response has reached a minimum level. Using a 10-ml syringe, extract a sample from the syringe port prior to the NDIR, and inject this sample into the NMO analyzer.

After the completion of the CO_2 purge, use the carrier gas bypass valve to pressurize the sample tank to approximately 1060 mm Hg absolute pressure with zero air.

4.3.3 Recovery of the Condensate Trap Sample. See Figure 25-10. Attach the ICV to the sample recovery system. With the sample recovery valve in a closed position, between vent and collect, and the flow control and ICV valves fully open, evacuate the manometer or gauge, the connecting tubing, and the ICV to 10 mm Hg absolute pressure. Close the flow-control and vacuum pump valves.

Begin auxiliary oxygen flow to the oxidation catalyst at a rate of 150 cc/min, then switch the four-way valve to the trap recovery position and the sample recovery valve to collect position. The system should now be set up to operate as indicated in Figure 25-10. After the manometer or pressure gauge begins to register a slight positive pressure, open the flow control valve. Adjust the flow-control valve to maintain atmospheric pressure in the system within 10 percent.

Now, remove the condensate trap from the dry ice, and allow it to warm to ambient temperature while monitoring the NDIR response. If after 5 minutes, the CO_2 concentration of the catalyst effluent is below 10,000 ppm, discontinue the auxiliary oxygen flow to the oxidation catalyst. Begin heating the trap by placing it in a furnace preheated to 200°C. Once heating has begun, carefully monitor the NDIR response to ensure that the catalyst effluent concentration does not exceed 50,000 ppm. Whenever the CO_2 concentration exceeds 50,000 ppm, supply auxiliary oxygen to the catalyst at the rate of 150 cc/min. Begin heating the tubing that connected the heated sample box to the condensate trap only after the CO_2 concentration falls below 10,000 ppm. This tubing may be heated in the same oven as the condensate trap or with an auxiliary heat source such as a heat gun. Heating temperature must not exceed 200°C. If a heat gun is used, heat the tubing slowly along its entire length from the upstream end to the downstream end, and repeat the pattern for a total of three times. Continue the recovery until the CO_2 concentration drops to less than 10 ppm as determined by syringe injection as described under the condensate trap CO_2 purge procedure, Section 4.3.2.

After the sample recovery is completed, use the carrier gas bypass valve to pressurize the ICV to approximately 1060 mm Hg absolute pressure with zero air.

4.4 Analysis. Before putting the NMO analyzer into routine operation, conduct an initial performance test. Start the analyzer, and perform all the necessary functions in order to put the analyzer into proper working order; then conduct the performance test according to the procedures established in Section 5.2. Once the performance test has been successfully completed and the CO_2 and NMO calibration response factors have been determined, proceed with sample analysis as follows:

4.4.1 Daily Operations and Calibration Checks. Before and immediately after the analysis of each set of samples or on a daily basis (whichever occurs first), conduct a calibration test according to the procedures established in Section 5.3. If the criteria of the daily calibration test cannot be met, repeat the NMO analyzer performance test (Section 5.2) before proceeding.

4.4.2 Operating Conditions. The carrier gas flow rate is 29.5 cc/min He and 2.2 cc/min O_2. The column oven is heated to 85°C. The order of elution for the sample from the column is CO, CH_4, CO_2, and NMO.

4.4.3 Analysis of Recovered Condensate Sample. Purge the sample loop with sample, and then inject the sample. Under the specified operating conditions, the CO_2 in the sample will elute in approximately 100 seconds. As soon as the detector response returns to baseline following the CO_2 peak, switch the carrier gas flow to backflush, and raise the column oven temperature to 195°C as rapidly as possible. A rate of 30°C/min has been shown to be adequate. Record the value obtained for the condensible organic material (C_{cm}) measured as CO_2 and any measured NMO. Return the column oven temperature to 85°C in preparation for the next analysis. Analyze each sample in triplicate, and report the average C_{cm}.

4.4.4 Analysis of Sample Tank. Perform the analysis as described in Section 4.4.3, but record only the value measured for NMO (C_{tm}).

4.5 Audit Samples. Analyze a set of two audit samples concurrently with any compliance samples and in exactly the same manner to evaluate the analyst's technique and the instrument calibration. The same analysts, analytical reagents, and analytical system shall be used for the compliance samples and the EPA audit samples; if this condition is met, auditing of subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both enforcement agencies.

Calculate the concentrations of the audit samples in ppm using the specified sample volume in the audit instructions. (NOTE: Indication of acceptable results may be obtained immediately by reporting the audit results in ppm and compliance results in ppm by telephone to the responsible enforcement agency.) Include the results of both audit samples, their identification numbers, and the analyst's name with the results of the compliance determination samples in appropriate reports to the EPA regional office or the appropriate enforcement agency during the 30-day period.

The concentration of the audit samples obtained by the analyst shall agree within 20 percent of the actual concentrations. Failure to meet the 20 percent specification may require retests until the audit problems are resolved. However, if the audit results do not affect the compliance or noncompliance status of the affected facility, the Administrator may waive the reanalysis requirement, further audits, or retests and accept the results of the compliance test. While steps are being taken to resolve audit analysis problems, the Administrator may also choose to use the data to determine the compliance or noncompliance of the affected facility.

5. Calibration and Operational Checks

Maintain a record of performance of each item.

5.1 Initial Performance Check of Condensate Recovery Apparatus. Perform these tests before the system is first placed in operation, after any shutdown of 6 months or more, and after any major modification of the system, or at the specified frequency.

5.1.1 Carrier Gas and Auxiliary O_2 Blank Check. Analyze each new tank of carrier gas or auxiliary O_2 with the NMO analyzer to check for contamination. Treat the gas cylinders as noncondensible gas samples, and analyze according to the procedure in Section 4.4.3. Add together any measured CH_4, CO, CO_2, or NMO. The total concentration must be less than 5 ppm.

5.1.2 Catalyst Efficiency Check. With a clean condensate trap installed in the recovery system, replace the carrier gas cylinder with the high level methane standard gas cylinder (Section 3.4.1). Set the four-port valve to the recovery position, and attach an ICV to the recovery system. With the sample recovery valve in vent position and the flow-control and ICV valves fully open, evacuate the manometer or gauge, the connecting tubing, and the ICV to 10 mm Hg absolute pressure. Close the flow-control and vacuum pump valves.

After the NDIR response has stabilized, switch the sample recovery valve from vent to collect. When the manometer or pressure gauge begins to register a slight positive pressure, open the flow-control valve. Keep the flow adjusted so that atmospheric pressure is maintained in the system within 10 percent. Continue collecting the sample in a normal manner until the ICV is filled to a nominal gauge pressure of 300 mm Hg. Close the ICV valve, and remove the ICV from the system. Place the sample recovery valve in the vent position, and return the recovery system to its normal carrier gas and normal operating conditions. Analyze the ICV for CO_2 using the NMO analyzer; the catalyst efficiency is acceptable if the CO_2 concentration is within 2 percent of the methane standard concentration.

5.1.3 System Performance Check. Construct a liquid sample injection unit similar in design to the unit shown in Figure 25-7. Insert this unit into the condensate recovery and conditioning system in place of a condensate trap, and set the carrier gas and auxiliary O_2 flow rates to normal operating levels. Attach an evacuated ICV to the system, and switch from system vent to collect. With the carrier gas routed through the injection unit and the oxidation catalyst, inject a liquid sample (See Sections 5.1.3.1 to 5.1.3.4) into the injection port. Operate the trap recovery system as described in Section 4.3.3. Measure the final ICV pressure, and then analyze the vessel to determine the CO_2 concentration. For each injection, calculate the percent recovery using the equation in Section 6.6.

The performance test is acceptable if the average percent recovery is 100 ± 10 percent with a relative standard deviation (Section 6.9) of less than 5 percent for each set of triplicate injections as follows:

5.1.3.1 50 ul Hexane.

5.1.3.2 10 ul Hexane.

5.1.3.3 50 ul Decane.

5.1.3.4 10 ul Decane.

5.2 Initial NMO Analyzer Performance Test. Perform these tests before the system is first placed in operation, after any shutdown longer than 6 months, and after any major modification of the system.

5.2.1 Oxidation Catalyst Efficiency Check. Turn off or bypass the NMO analyzer reduction catalyst. Make triplicate injections of the high level methane standard (Section 3.4.1). The oxidation catalyst operation is acceptable if the FID response is less than 1 percent of the injected methane concentration.

5.2.2 Reduction Catalyst Efficiency Check. With the oxidation catalyst unheated or bypassed and the heated reduction catalyst bypassed, make triplicate injections of the high level methane standard (Section 3.4.1). Repeat this procedure with both catalysts operative. The reduction catalyst operation is acceptable if the response under both conditions agree within 5 percent.

5.2.3 Analyzer Linearity Check and NMO Calibration. While operating both the oxidation and reduction catalysts, conduct a linearity check of the analyzer using the propane standards specified in Section 3.4.2. Make triplicate injections of each calibration gas, and then calculate the average response factor (area/ppm C) for each gas, as well as the overall mean of the response factor values. The instrument linearity is acceptable if the average response factor of each calibration gas is within 2.5 percent of the overall mean value and if the relative standard deviation (Section 6.9) for each set of triplicate injections is less than 2 percent. Record the overall mean of the propane response factor values as the NMO calibration response factor (RF_{NMO}).

Repeat the linearity check using the CO_2 standards specified in Section 3.4.3. Make triplicate injections of each gas, and then calculate the average response factor (area/ppm C) for each gas, as well as the overall mean of the response factor values. Record the overall mean of the response factor values as the CO_2 calibration response factor (RF_{CO_2}). Linearity is acceptable if the average response factor of each calibration gas is within 2.5 percent of the overall mean value and if the relative standard deviation for each set of triplicate injections is less than 2 percent. The RF_{CO_2} must be within 10 percent of the RF_{NMO}.

5.2.4 System Performance Check. Check the column separation and overall performance of the analyzer by making triplicate injections of the calibration gases listed in Section 3.4.4. The analyzer performance is acceptable if the measured NMO value for each gas (average of triplicate injections) is within 5 percent of the expected value.

5.3 NMO Analyzer Daily Calibration.

5.3.1 CO_2 Response Factor. Inject triplicate samples of the high level CO_2 calibration gas (Section 3.4.3). and calculate the average response factor. The system operation is adequate if the calculated response factor is within 5 percent of the RF_{CO_2} calculated during the initial performance test (Section 5.2.3). Use the daily response factor (DFR_{CO_2}) for analyzer calibration and the calculation of measured CO_2 concentrations in the ICV samples.

5.3.2 NMO Response Factors. Inject triplicate samples of the mixed propane calibration cylinder (Section 3.4.4.1), and calculate the average NMO response factor. The system operation is adequate if the calculated response factor is within 5 percent of the RF_{NMO} calculated during the initial performance test (Section 5.2.4). Use the daily response factor (DRF_{NMO}) for analyzer calibration and calculation of NMO concentrations in the sample tanks.

5.4 Sample Tank and ICV Volume. The volume of the gas sampling tanks used must be determined. Determine the tank and ICV volumes by weighing them empty and then filled with deionized distilled water; weigh to the nearest 5 g, and record the results. Alternatively, measure the volume of water used to fill them to the nearest 5 ml.

6. Calculations

All equations are written using absolute pressure; absolute pressures are determined by adding the measured barometric pressure to the measured gauge or manometer pressure.

6.1 Nomenclature.

C = TGNMO concentration of the effluent, ppm C equivalent.
C_c = Calculated condensible organic (condensate trap) concentration of the effluent, ppm C equivalent.
C_{cm} = Measured concentration (NMO analyzer) for the condensate trap ICV, ppm CO_2.
C_t = Calculated noncondensible organic concentration (sample tank) of the effluent, ppm C equivalent.
C_{tm} = Measured concentration (NMO analyzer) for the sample tank, ppm NMO.
F = Sampling flow rate, cc/min.
L = Volume of liquid injected, ul.
M = Molecular weight of the liquid injected, g/g-mole.
m_c = TGNMO mass concentration of the effluent, mg C/dsm^3.
N = Carbon number of the liquid compound injected (N = 12 for decane, N = 6 for hexane).
P_f = Final pressure of the intermediate collection vessel, mm Hg absolute.
P_b = Barometric pressure, cm Hg.
P_{ti} = Gas sample tank pressure before sampling, mm Hg absolute.
P_t = Gas sample tank pressure after sampling, but before pressurizing, mm Hg absolute.
P_{tf} = Final gas sample tank pressure after pressurizing, mm Hg absolute.
T_f = Final temperature of intermediate collection vessel, °K.
T_{ti} = Sample tank temperature before sampling, °K.
T_t = Sample tank temperature at completion of sampling, °K.
T_{tf} = Sample tank temperature after pressurizing, °K.
V = Sample tank volume, m^3.
V_t = Sample train volume, cc.
V_v = Intermediate collection vessel volume, m^3.
V_s = Gas volume sampled, dsm^3.
n = Number of data points.
q = Total number of analyzer injections of intermediate collection vessel during analysis (where k = injection number, 1 ... q).
r = Total number of analyzer injections of sample tank during analysis (where j = injection number, 1 ... r).
x_i = Individual measurements.
x = Mean value.
ρ = Density of liquid injected, g/cc.
θ = Leak check period, min.
ΔP = Allowable pressure change, cm Hg.

6.2 Allowable Pressure Change. For the pretest leak check, calculate the allowable pressure change:

$$\Delta P = 0.01 \frac{FP_b \theta}{V_t} \qquad \text{Eq. 25-1}$$

6.3 Sample Volume. For each test run, calculate the gas volume sampled:

$$V_s = 0.3857\ V \left[\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}} \right] \qquad \text{Eq. 25-2}$$

6.4 Noncondensible Organics. For each sample tank, determine the concentration of nonmethane organics (ppm C):

$$C_t = \left[\frac{\dfrac{P_{tf}}{T_{tf}}}{\dfrac{P_t}{T_t} - \dfrac{P_{ti}}{T_{ti}}} \right] \left[\frac{1}{r} \sum_{j=1}^{r} C_{tm_j} \right] \qquad \text{Eq. 25-3}$$

6.5 Condensible Organics. For each condensate trap, determine the concentration of organics (ppm C):

$$C_c = 0.3857 \frac{V_v\ P_f}{V_s\ T_f} \left[\frac{1}{q} \sum_{k=1}^{q} C_{cm_k} \right] \qquad \text{Eq. 25-4}$$

6.6 TGNMO. To determine the TGNMO concentration for each test run, use the following equation:

$$C = C_t + C_c \qquad \text{Eq. 25-5}$$

6.7 TGNMO Mass Concentration. To determine the TGNMO mass concentration as carbon for each test run, use the following equation:

$$m_c = 0.4993\ C \qquad \text{Eq. 25-6}$$

6.8 Percent Recovery. To calculate the percent recovery for the liquid injections to the condensate recovery and conditioning system use the following equation:

$$\text{Percent recovery} = 1.604 \frac{M}{L} \frac{V_v}{\rho} \frac{P_f}{T_f} \frac{C_{cm}}{N} \qquad \text{Eq. 25-7}$$

6.9 Relative Standard Deviation.

$$RSD = \frac{100}{\bar{x}}\sqrt{\frac{\Sigma(x_i - \bar{x})^2}{n - 1}} \qquad \text{Eq. 25-8}$$

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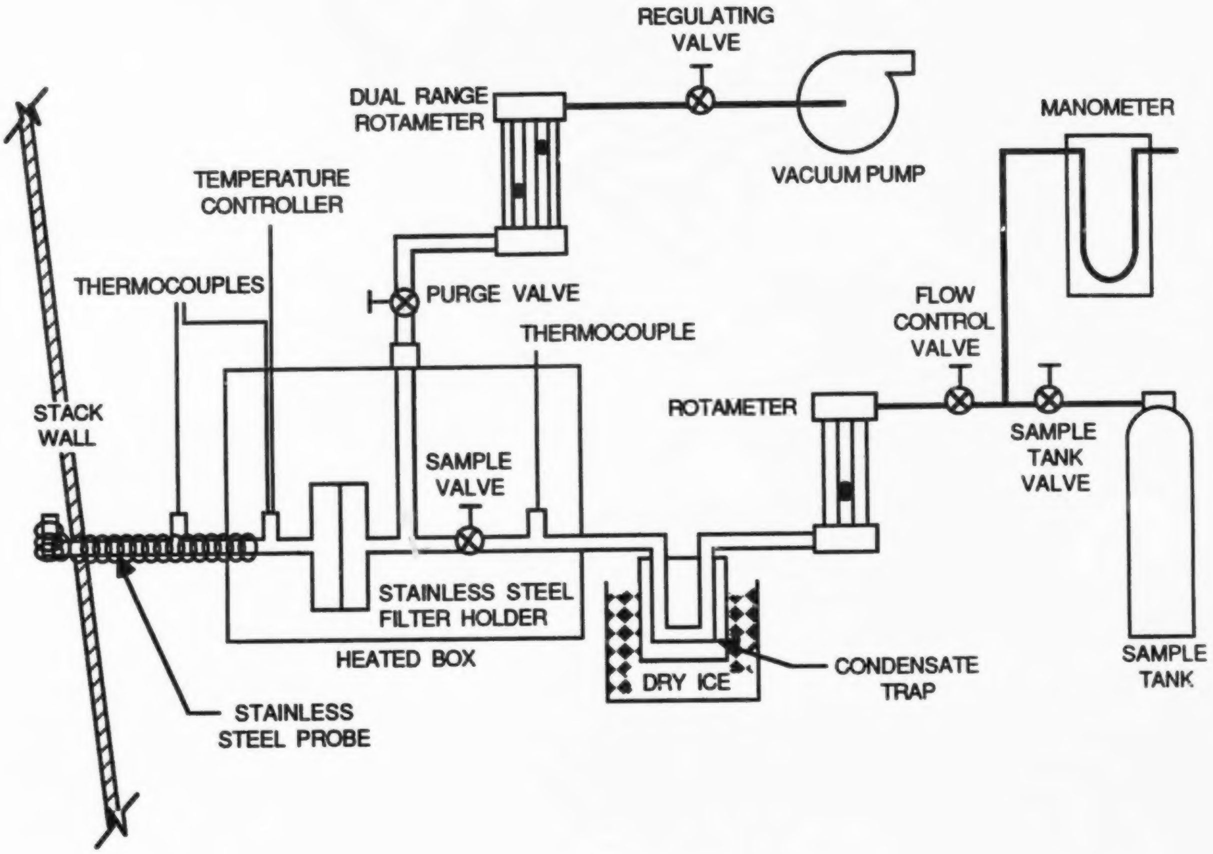
REGULATING VALVE
DUAL RANGE ROTAMETER
VACUUM PUMP
MANOMETER
TEMPERATURE CONTROLLER
THERMOCOUPLES
PURGE VALVE
THERMOCOUPLE
FLOW CONTROL VALVE
ROTAMETER
STACK WALL
SAMPLE VALVE
SAMPLE TANK VALVE
STAINLESS STEEL FILTER HOLDER
HEATED BOX
DRY ICE
CONDENSATE TRAP
SAMPLE TANK
STAINLESS STEEL PROBE

Figure 25-1. Sampling train.

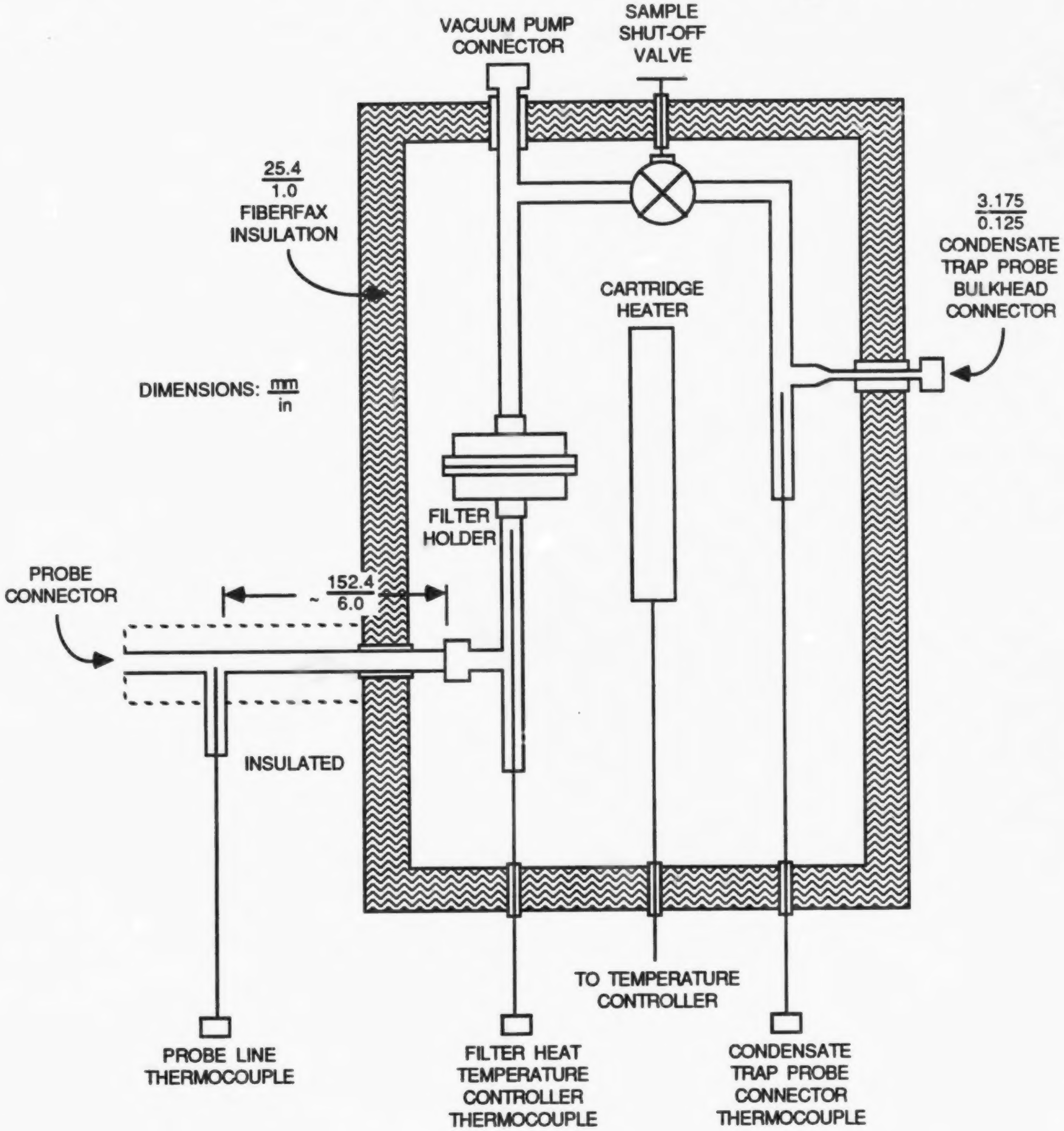
VACUUM PUMP CONNECTOR
SAMPLE SHUT-OFF VALVE
25.4 / 1.0
FIBERFAX INSULATION
3.175 / 0.125
CONDENSATE TRAP PROBE BULKHEAD CONNECTOR
CARTRIDGE HEATER
DIMENSIONS: mm / in
FILTER HOLDER
PROBE CONNECTOR
~ 152.4 / 6.0
INSULATED
TO TEMPERATURE CONTROLLER
PROBE LINE THERMOCOUPLE
FILTER HEAT TEMPERATURE CONTROLLER THERMOCOUPLE
CONDENSATE TRAP PROBE CONNECTOR THERMOCOUPLE

Figure 25-2. Out-of-stack filter box.

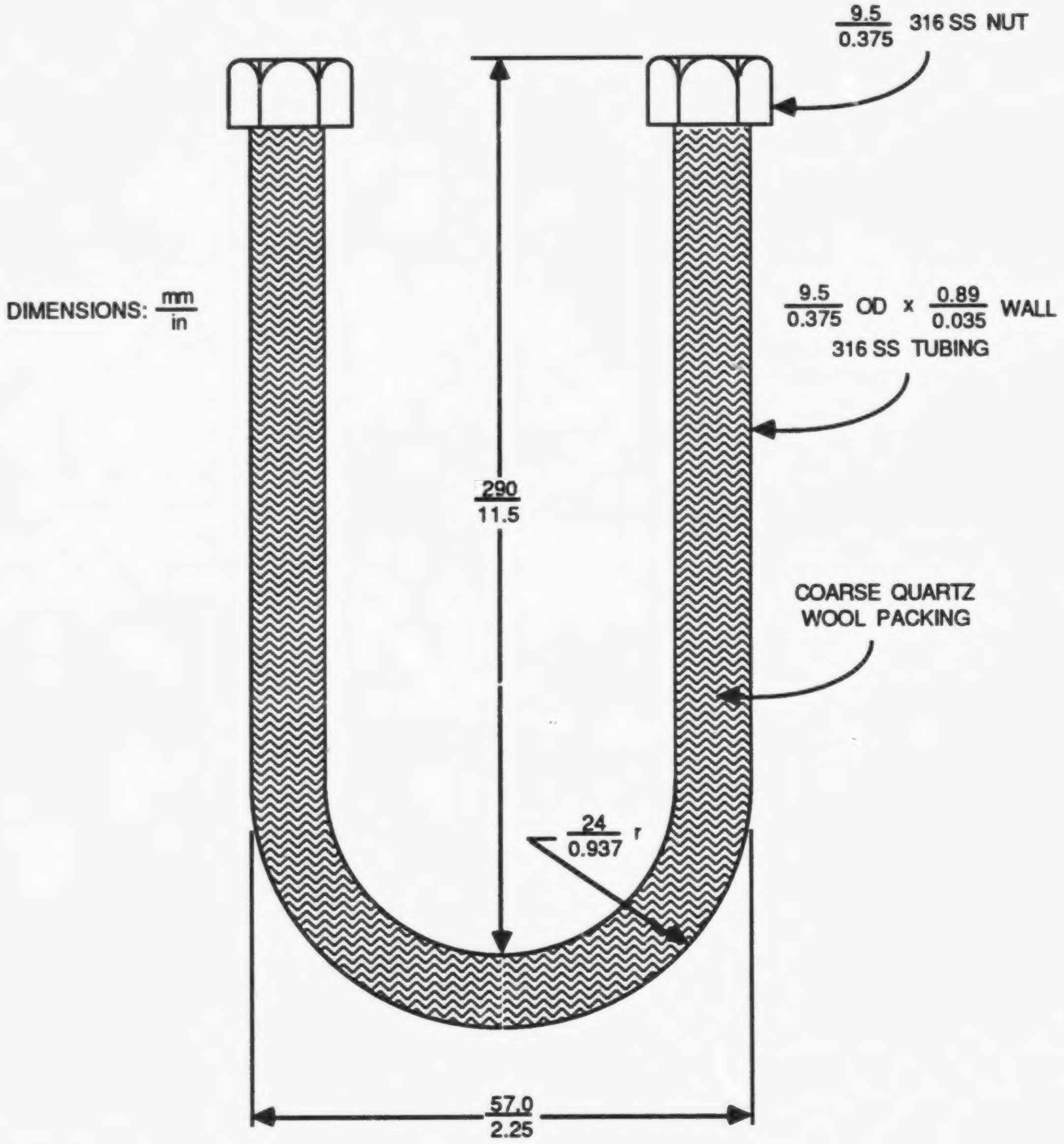
$\frac{9.5}{0.375}$ 316 SS NUT
DIMENSIONS: $\frac{mm}{in}$
$\frac{9.5}{0.375}$ OD x $\frac{0.89}{0.035}$ WALL
316 SS TUBING
$\frac{290}{11.5}$
COARSE QUARTZ
WOOL PACKING
$\frac{24}{0.937}$ r
$\frac{57.0}{2.25}$

Figure 25-3. Condensate trap.

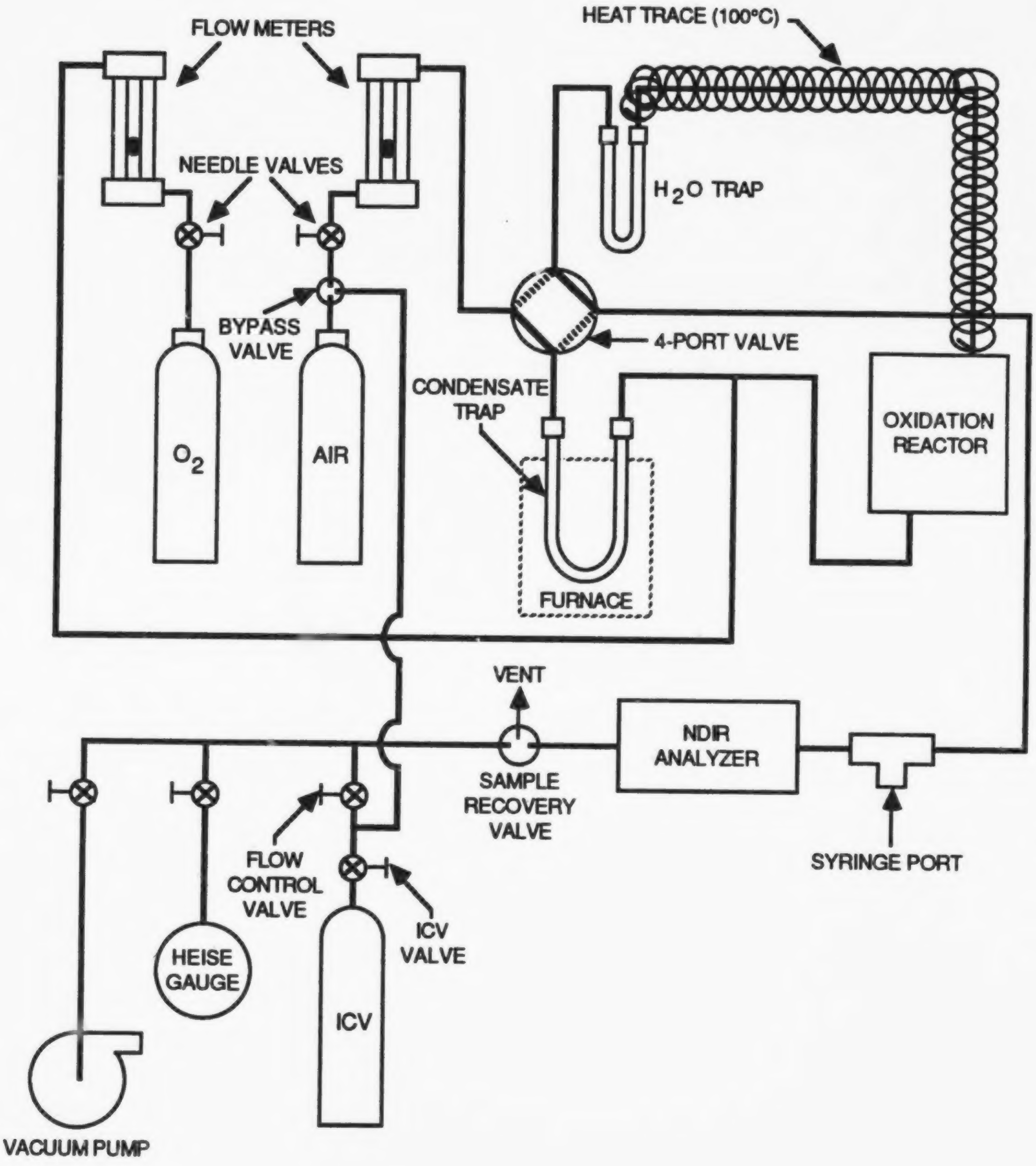
FLOW METERS
HEAT TRACE (100°C)
NEEDLE VALVES
H$_2$O TRAP
BYPASS VALVE
4-PORT VALVE
CONDENSATE TRAP
O$_2$
AIR
OXIDATION REACTOR
FURNACE
VENT
NDIR ANALYZER
SAMPLE RECOVERY VALVE
SYRINGE PORT
FLOW CONTROL VALVE
ICV VALVE
HEISE GAUGE
ICV
VACUUM PUMP

Figure 25-4. Condensate recovery system.

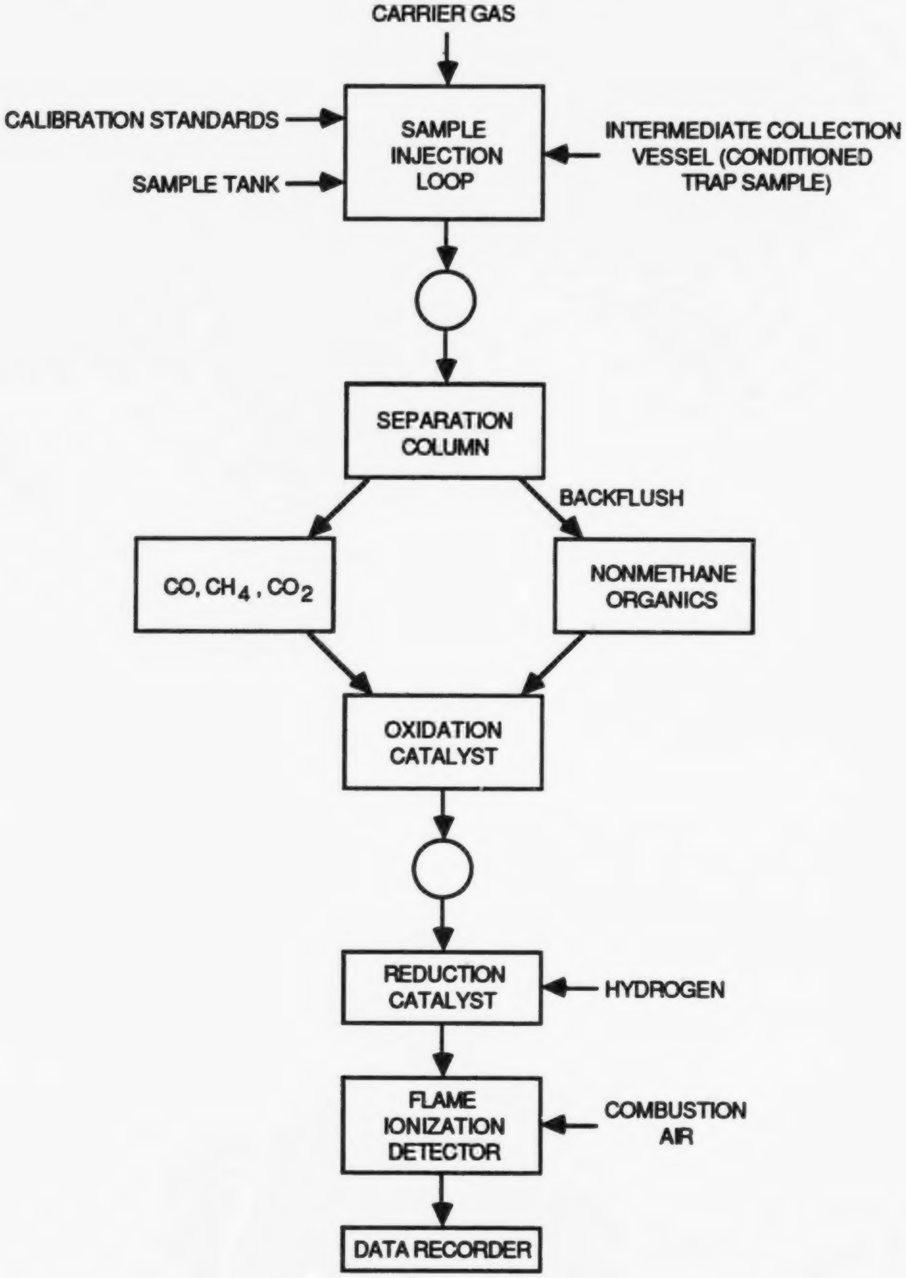
CARRIER GAS
CALIBRATION STANDARDS
SAMPLE TANK
SAMPLE INJECTION LOOP
INTERMEDIATE COLLECTION VESSEL (CONDITIONED TRAP SAMPLE)
SEPARATION COLUMN
BACKFLUSH
CO, CH4, CO2
NONMETHANE ORGANICS
OXIDATION CATALYST
REDUCTION CATALYST
HYDROGEN
FLAME IONIZATION DETECTOR
COMBUSTION AIR
DATA RECORDER

Figure 25-5. Simplified schematic of nonmethane organic (NMO) analyzer.

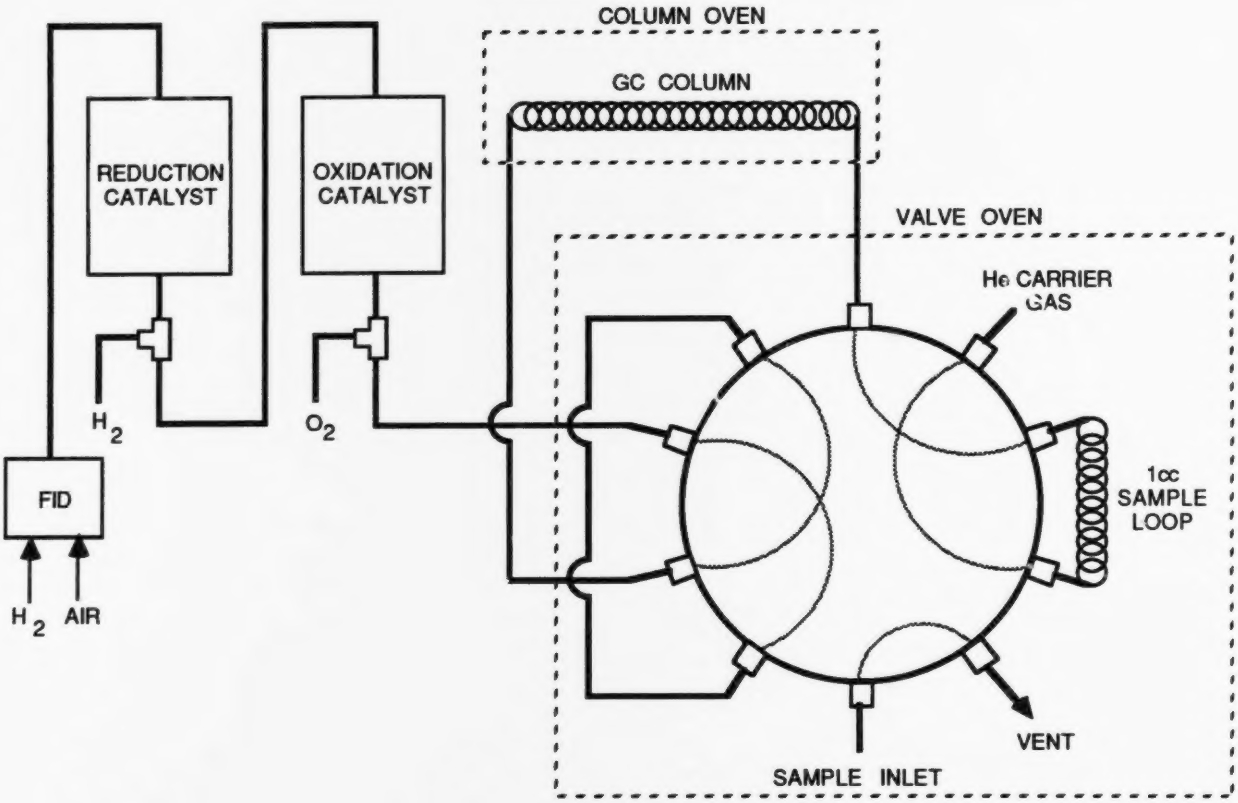
COLUMN OVEN
GC COLUMN
REDUCTION CATALYST
OXIDATION CATALYST
VALVE OVEN
He CARRIER GAS
H_2
O_2
FID
H_2
AIR
1cc SAMPLE LOOP
VENT
SAMPLE INLET

Figure 25-6. Nonmethane organic (NMO) analyzer.

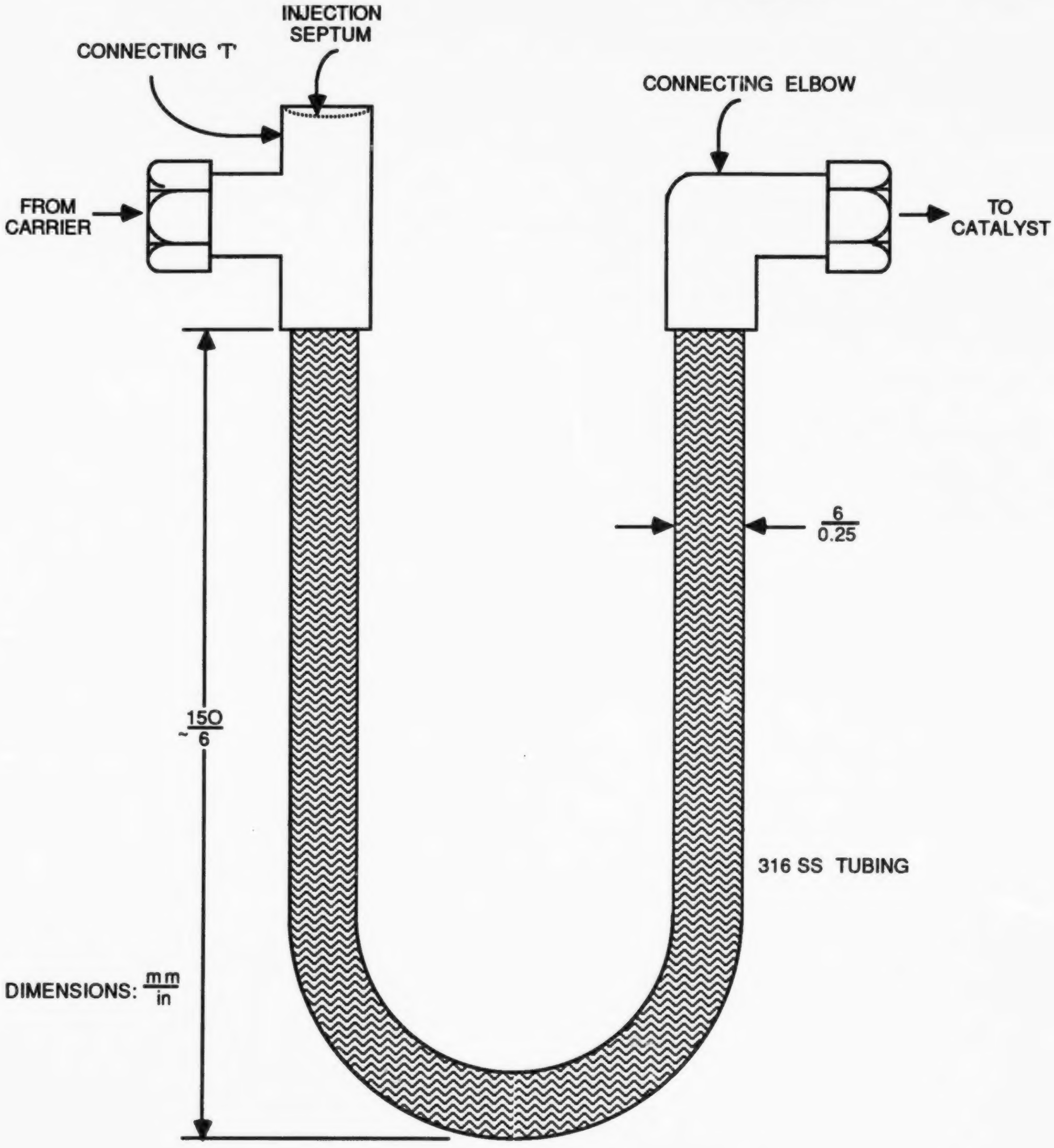
INJECTION SEPTUM
CONNECTING 'T'
CONNECTING ELBOW
FROM CARRIER
TO CATALYST
$\frac{6}{0.25}$
$\sim\frac{150}{6}$
316 SS TUBING
DIMENSIONS: $\frac{mm}{in}$

Figure 25-7. Liquid sample injection unit.

VOLATILE ORGANIC CARBON

FACILITY ____________ SAMPLE LOCATION ____________
LOCATION ____________ OPERATOR ____________
DATE ____________ RUN NUMBER ____________
TANK NUMBER ____________ TRAP NUMBER ____________ SAMPLE ID NUMBER ____________

	TANK VACUUM, mm Hg		cm Hg	BAROMETRIC PRESSURE, mm Hg	AMBIENT TEMPERATURE, °C
PRETEST (MANOMETER)		(GAUGE)			
POST TEST (MANOMETER)		(GAUGE)			

LEAK RATE cm Hg / 10 min

PRETEST ____________

TIME CLOCK/SAMPLE	VACUUM cm Hg	FLOWMETER SETTING	COMMENTS

Figure 25-8. Example field data form.

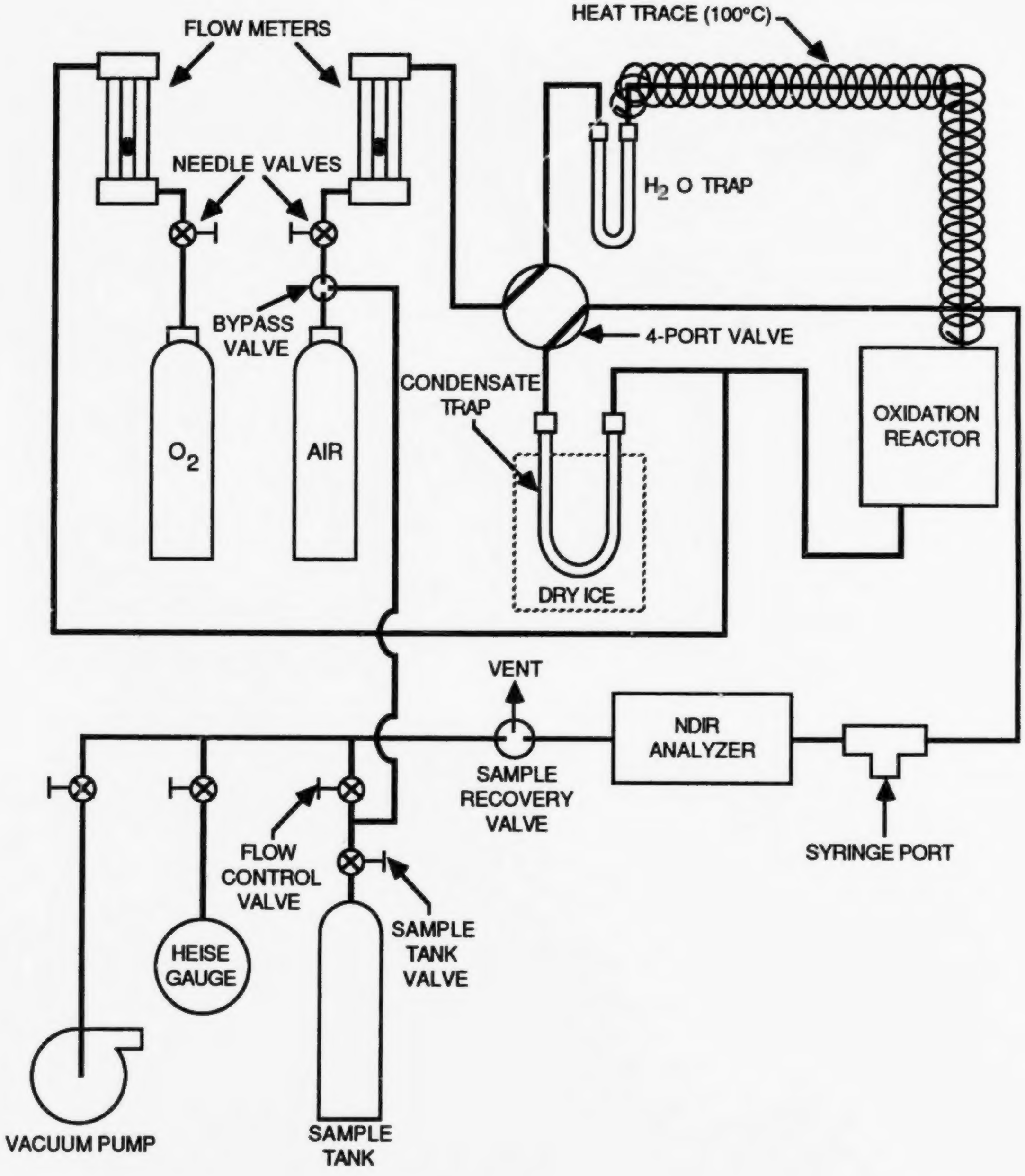
HEAT TRACE (100°C)
FLOW METERS
NEEDLE VALVES
H_2O TRAP
BYPASS VALVE
4-PORT VALVE
CONDENSATE TRAP
O_2
AIR
OXIDATION REACTOR
DRY ICE
VENT
NDIR ANALYZER
SAMPLE RECOVERY VALVE
SYRINGE PORT
FLOW CONTROL VALVE
HEISE GAUGE
SAMPLE TANK VALVE
VACUUM PUMP
SAMPLE TANK

Figure 25-9. Condensate recovery system, CO_2 purge.

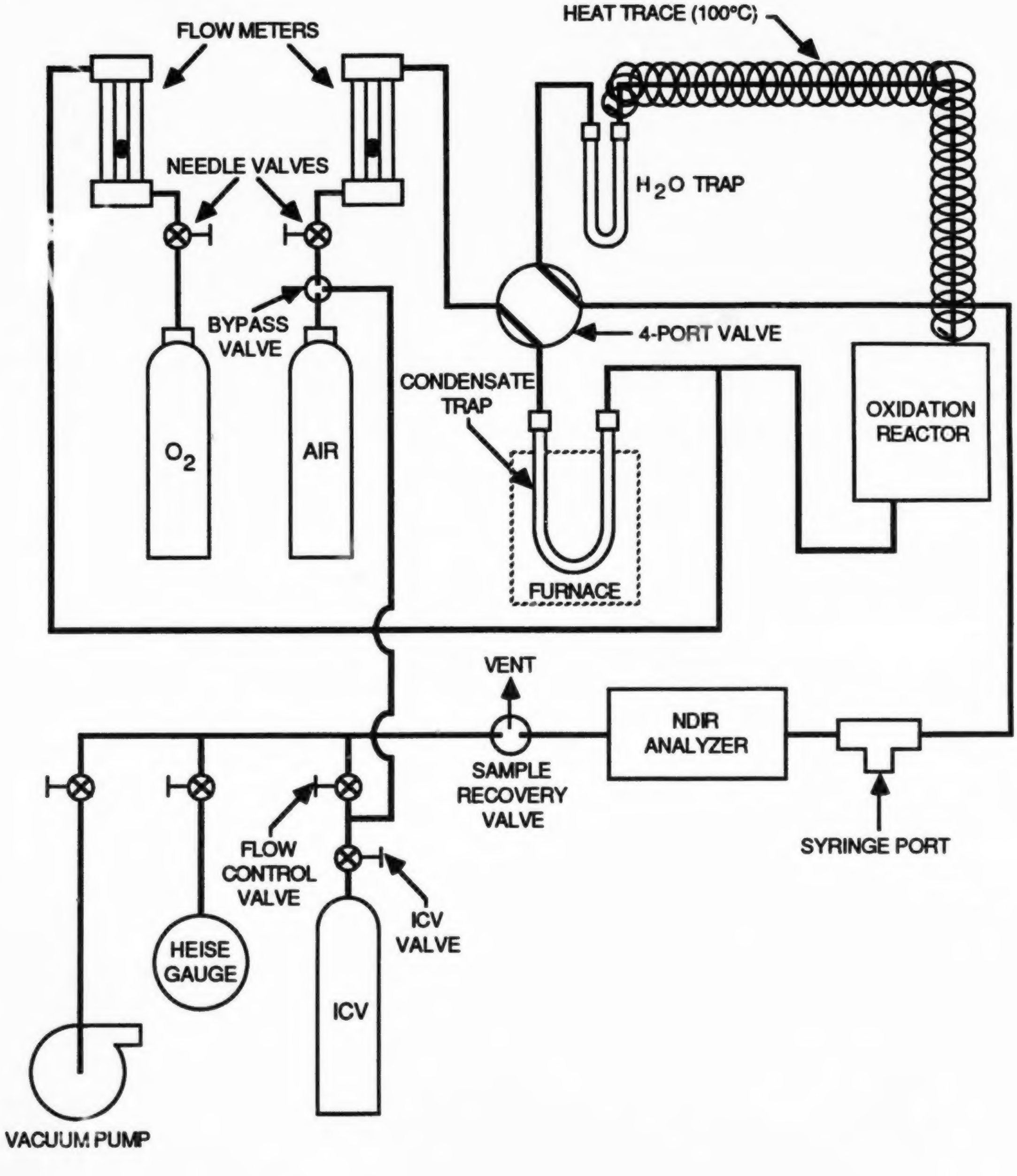
HEAT TRACE (100°C)
FLOW METERS
NEEDLE VALVES
H2O TRAP
BYPASS VALVE
4-PORT VALVE
CONDENSATE TRAP
O2
AIR
OXIDATION REACTOR
FURNACE
VENT
SAMPLE RECOVERY VALVE
NDIR ANALYZER
SYRINGE PORT
FLOW CONTROL VALVE
ICV VALVE
HEISE GAUGE
ICV
VACUUM PUMP

Figure 25-10. Condensate recovery system, collection of trap organics.

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